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Application of carbon nanotube filled silicone rubber composite in stress measurement during ramped loading with low compression speed

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1. Introduction

It is widely reported that conductive polymer composites can demonstrate a piezoresistive behavior [1-9]. Therefore, this kind of material has the potential to be used to develop flexible stress sensor [10–14]. Recently, there are more and more researches on the changes in the electrical resistance of the composite under applied stress or strain, including the instantaneous piezoresistivity (i.e. the relation between the resistance and the stress at the moment immediately after compression) [15–17], the resistance relaxation (i.e. the changes in the electrical resistance of the composite during the stress relaxation) [18-22], the resistance creep (i.e. the variations in the electrical resistance of the composite during the creep) [23,24], and the quasi-steady piezoresistivity (i.e. the piezoresistivity after the compression is applied on the sample for a long time (the resistance tends to be stable over time)) [25-27]. As for the instantaneous piezoresistivity and the quasi-steady piezoresistivity, both the stress and the strain are independent of time. For the resistance relaxation, the stress changes over time whereas the strain is invariant over time. For the resistance creep, the strain changes over time whereas the stress is invariant over time. In many engineering applications, the composite is usually under a "ramped loading", during which both the strain and the stress of the composite vary over time. The relation between the stress and the

ABSTRACT

To realize the stress measurement during the ramped loading with low compression speed (<1 mm/min), the piezoresistive responses of carbon nanotube filled silicone rubber composite to ramped loading are studied. If the conductive phase content of the composite is high, the piezoresistive curve is not monotonic. If the conductive phase content is low, the electrical resistance increases monotonically with the increase of the compressive stress, and the changing rate of the resistance increases with the increase of the compression speed. The phenomena are explained by analyzing the changes in the inner structure of the composite. A method to realize the conversion from the resistance of the composite to the stress during the ramped loading is proposed. The feasibility of using the piezoresistivity of the composite to measure the stress during the ramped loading with low compression speed is verified preliminarily.

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electrical resistance of the composite during the ramped loading is defined as "piezoresistive response to ramped loading".

Recently, there are some researches on the "piezoresistive response to ramped loading" [28-33]. Yoshimura et al. [29] studied the effect of the tensile strain on the electrical resistivity of carbon microcoil filled silicone rubber composites. Their measurements were carried out by using the tensile tester at a constant speed of 10 mm/min. They found that the resistivity increases with the increase of the strain. Knite et al. [30] researched the piezoresistive effect of multi-wall carbon nanotube filled polyisoprene composites at a strain velocity of 6.67×10^{-5} m/s. Zhou et al. [31] investigated the piezoresistivity for carbon black filled poly(methylvinylsilioxane) vulcanizates at a speed of 0.5 mm/min. Das et al. [32] researched the effect of axial stretching on electrical resistivity of short carbon fiber and carbon black filled conductive rubber composites. In their research, the strain rates are 0.1 mm/min, 1 mm/min, and 10 mm/min. They found that the tensile strain increases with the decrease of the strain rate under the same stress and the resistivity increases with the increase of the strain rate under the same tensile strain.

According to the aforementioned introduction, we can see that most of the previous researches on the piezoresistive response to ramped loading were developed under a constant speed. Only a few researches involved the piezoresistive responses to ramped loading under different strain rates in tension state [32]. However, there are seldom researches on the influence of compression speed on the piezoresistive response to ramped loading in compression state. As the composite is viscoelastic, the piezoresistive response of the composite to ramped loading in compression state

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is very complicated. It is not only dependent on the conductive phase content, but also related to the compression speed. Therefore, it is urgently needed to study the piezoresistive response to ramped loading under different compression speeds. In our previous research, the sample is compressed at a very high speed, and the resistance shows a sudden increase [19,22]. However, we found that the piezoresistive curve of the composite is not monotonic when the compression speed is relatively low. This phenomenon will bring difficulty to the development of sensor. (To develop a sensor based on conductive polymer composite to monitor the stress during ramped loading, a monotonic piezoresistivity of the composite is needed) In this paper, the relations between the stresses and the electrical resistances of carbon nanotube filled silicone rubber composites under ramped loading with the compression speeds lower than 1 mm/min are studied. The importance and new contribution of this work include: (1) By lowering the conductive phase content of the composite, a monotonic piezoresistivity under the low compression speed can be obtained. (2) For the composite with monotonic piezoresistivity, the changing rate of the resistance of the composite increases with the increase of the compression speed. Based on this result, the feasibility of using the changing rate of the resistance to measure the compression speed has been verified. (3) A method to realize the conversion from the electrical resistance of the composite to the stress during the ramped loading is proposed and verified.

2. Experimental

Carbon nanotube is used as the conductive phase. Room temperature vulcanized liquid silicone rubber is used as the insulating phase. The characteristics of silicone rubber and carbon nanotube are shown in Table 1. Carbon nanotubes are dispersed into silicone rubber by using solution mixing method. Ethyl silicate is used as a crosslinker. Hexane is used as a solvent to mix carbon nanotubes into silicone rubber. Mechanical stirring along with ultrasonic vibration is used for better filler dispersion. After 3 h of

Table 1

Characteristics of silicone rubber and carbon nanotube.

| Carbon nanotube | Resistivity | Aspect ratio | рН |
|-----------------|---------------------|--------------|---------------------|
| | <1 Ω cm | 330 | >95% |
| Silicone | Dielectric constant | Hardness | Dielectric strength |
| Rubber | 3.0 | 35 Shore | 15 kV/mm |

vigorous mixing, the solvent is evaporated at 40 °C. The viscous mixture is vulcanized between the two electrodes at room temperature for around 60 h. Then, the sample with sandwich structure $(8 \text{ mm} \times 8 \text{ mm} \times 5 \text{ mm})$ is completed.

The experimental setup is shown in Fig. 1. The sample with the sandwich structure is placed between the movable head and the fix platform of the material testing machine (WDW-5kN). The sample is compressed from 0 to 0.6 MPa by the downward movement of the movable head. The compression speeds are 0.1, 0.3, 0.5, 0.8 and 1 mm/min. The values of the stress and the strain are recorded by the material testing machine. The electrical resistance is measured through the digital multi-meter (Agilent 34410A) at room temperature.

3. Results and discussion

Twelve specimens are fabricated for the composite with the same conductive phase content. Each test is repeated for twelve times. The maximum deviation among the electrical resistances of the specimens with the same conductive phase content under the same stress is less than 3%. The experimental results show that the piezoresistivity under the ramped loading with low compression speed (0–1 mm/min) is dependent on the conductive phase content. If the mass ratio of carbon nanotube to silicone rubber is higher than 0.06, the piezoresistive curve is non-monotonic. If the mass ratio is lower than 0.06, the piezoresistive curve is monotonically increasing. To present the results clearly and concisely, the piezoresistivities of the composites with two typical mass ratios (0.04 and 0.12) are chosen to be described in detail in Fig. 1. "C"



Fig. 1. Piezoresistivities of the composites with different conductive phase contents.

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