



Pressure and temperature induced electrical resistance change in nano-carbon/epoxy composites



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ABSTRACT

In this study, we investigate the changes of electrical resistance of the carbon black (CB) and carbon nanotube (CNT) filled epoxy composites upon compression, swelling and temperature variation. For all samples we observe a decrease of electrical resistance under compression, while an increase of electrical resistance is found upon swelling due to water absorption. With the same volumetric change, the CB/epoxy composites have a more pronounced change of electrical resistance than the CNT/epoxy composites. The influence of temperature variations on the overall electrical resistance is somewhat more complex. At low temperatures, electrical resistance clearly decreases with increasing temperature, mainly owing to the thermal fluctuation induced tunneling conduction mechanism. At high temperatures, electrical resistance decreases much slower and may even increase due to a more prominent thermal expansion effect. The thermal expansion of the epoxy matrix leads to a decrease of tunneling conduction. Without the contribution of the thermal expansion, the sensitivity and linearity of electrical resistance change upon temperature variations is higher.

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1. Introduction

Nanoparticles such as carbon black (CB), and carbon nanotubes (CNTs) are used to introduce moderate to high electrical conductivity in otherwise isolating polymers, with extremely low volume contents [1,2]. The measured electrical resistance of a conductive nano-carbon/polymer composite is sensitive to various influences beyond those typically observed in homogeneous materials like metal alloys. It is observed that electrical properties are influenced through pressure, temperature, and deformation [3,4]. Most research has been found to address unidirectional compression, tension or hydrostatic compression of the composites [5–7] and shows that tensile strains could cause an increase in the electrical resistance of composites, while compressive strains lead to a decrease. However, we know from our own previous research (affiliation “b”) that with regard to high aspect ratio particles, this is not always the case and the situation seems rather more complex. It should be noted that in the cases of low CNT/CB filler

fractions (especially <1 wt.%), the electrical resistance of polymer matrix is dominant, so that little change of the electrical resistance of nano-carbons (e.g. temperature variation induced [8]) has only minute influence on the change of total electrical resistance of polymer composites. Regarding the effect of temperature, change of electrical resistance in the nano-carbon/polymer composites under temperature variation has also been widely reported [9–12]. This endows these composite materials with the possibility of harnessing extremely small and local pressure and temperature sensors for a great number of technical applications.

For this purpose, it is very important to be able to characterize or predict the electrical resistance change in the nano-carbon/polymer composites occurring with variation of pressure and temperature. It is already well known that tunneling conduction mechanisms play an important role in the electrical resistance change of a conductive nano-carbon/polymer composite. Some authors [13–16] reported a slower-than-linear decrease of electrical resistance with increasing temperature in conductive filler based polymer composites. However, other authors observed in nano-carbon/polymer composites an increase of electrical resistance with increasing temperature [17,18]. The causes of this apparent inconsistency deserve a closer look. It is the authors' perception that many apparently conflicting observations are the

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result of the large number of influencing phenomena and their relative importance in various cases. However, this is difficult to resolve in a material class where not only properties such as electrical conductivity, but also the properties of the constituents themselves, their fraction, and morphology are different.

In this work, the electrical resistance changes with compression, swelling upon absorption of water, and temperature variations are investigated, as these are some of the technologically most important aspects. Carbon nanoparticle/epoxy composites with various filler volume fractions of CNTs and CB, as well as different aspect ratios (ratio of length to diameter) of CNTs, are prepared and investigated. This study aims to contribute to a better understanding of the influence of the thermal expansion induced widening of tunneling gaps on the tunneling conduction in nano-carbon/epoxy composites.

2. Experimental

2.1. Materials

Two epoxy systems are used: (1) Araldite LY 556/Aradur 917/Accelerator DY070 (LY556 in short); (2) RIM 135/RIMH 137 (RIM135 in short). The viscosity of the two reactive mixtures (resin + hardener/accelerator) at 25 °C are around 750 mPa s and 300 mPa s, respectively. The bulk modulus of the LY556 is about 3200 MPa, while that of the RIM135 is about 2900 MPa, according to the datasheet of the manufacturer. Printex® XE2 carbon black (CB), and the multi wall carbon nanotubes (Baytubes® C150P (C150P) and Nanocyl™ NC7000 (NC7000)) were used as nano-carbon fillers. The Printex® XE2 CB has particle diameter of about 35 nm. The length and diameter of carbon nanotubes as measured in [19,20] are shown in Table 1. The measured lengths of the carbon nanotubes are different from the lengths reported in the manufacturers' datasheets.

A master batch (pre-mixture of resin and nano-particles) was produced using a high shear mixing process involving a lab-scale three-roll-mill (Exakt 120E). Details of the production process

Table 1
The average diameter and length of C150P and NC7000 carbon nanotubes.

	From literature		
	Diameter (nm)	Length (nm)	Aspect ratio
Baytubes® C150P ¹⁹	10.5	770	73
Baytubes® C150P ²⁰	11.6	610	52.7
Nanocyl™ NC7000 ¹⁹	10.0	1341	134

can be found elsewhere [21]. A further mixing of the master batch, hardener and accelerator was carried out with intensive manual-mixing for 10 min, followed by curing in a panel heated computer-controlled oven. The curing of LY556 and RIM135 followed their standard curing cycles. We then cut and polished the cured composite specimens to the desired dimensions. Since the dispersion of nano-fillers is a critical parameter, we kept preparation of the dispersion and subsequent processing conditions identical as much as possible. Representative TEM and SEM images can be found here [22,23].

2.2. Hydrostatic compression tests

A modified tablet press (as shown in Fig. 1b) was used to carry out hydrostatic compression tests. First, we attached the samples with desired dimensions ($5 \times 4 \times 3$ mm) to copper leads at the 4×3 mm faces using conductive silver paint. After drying, the samples were immersed into soft melted wax, and excess wax was removed after solidification. The final thickness of wax layer on the samples amounted to about 0.5–1.0 mm. Then, the wax-wrapped samples were immersed into RIM135 resin, before it was cured in the cavity of the IR tablet press. The cured sample in its final state is around 20 mm in height and 12 mm in diameter (Fig. 1a).

The samples were slightly moved inside the tablet press to avoid sticking. It should be noted that the top metal disk of the pill press has a small slot which only allows the two insulated copper leads passing through. Then, the IR tablet press with cylinder sample inside was transferred to a hot press, just fitting into the gap between its top plate and bottom plate. The temperature of both top and bottom plates were set at 55 °C to melt and further soften the wax surrounding the sample. The pressure was constant throughout the molten wax, making for a good approximation of hydrostatic compression. Each electrical resistance measurement was done 30 s after applying a pre-load. We recorded the resistance data with a Keithley 2601A under 5 V applied voltage. The highest pressure on the top surface of the epoxy cylinder approximated 295 MPa, derived from a maximum load of 33 kN on a 112 mm² area.

2.3. Water absorption

Water absorption tests were performed with samples (~ 40 mm \times 5 mm \times 3 mm) of the C150P/LY556 composites. The initial weight of every sample was measured in the ambient environment. Then, the samples were submerged in distilled water for

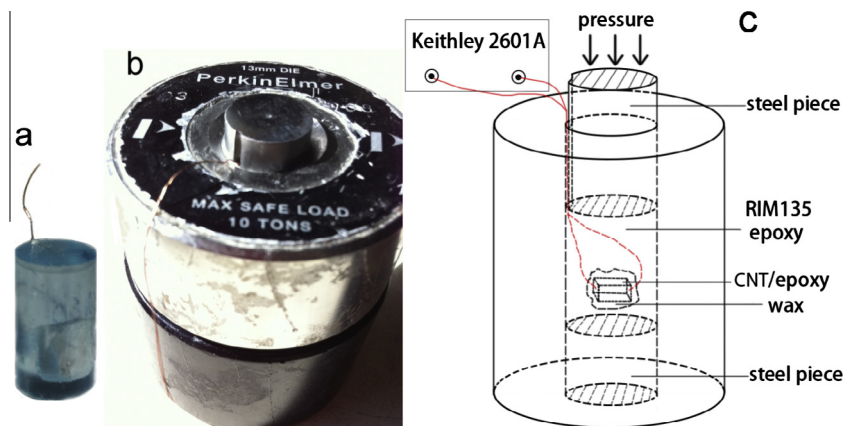


Fig. 1. (a) Test cylinder with small sample wrapped by wax and further by cured epoxy, final sample size: 20 mm \times \varnothing 12 mm; (b) self-modified IR tablet press for making sample cylinders and the testing apparatus. (c) Schematic drawing of (b) and the measuring design.

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