NANO-CARBON FILLERS IN A RESIN MATRIX: ELECTRICAL PROPERTIES

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1 Introduction

Polymer nanocomposites [1] have attracted a lot of attention in the last few years due to their enhanced properties at low weight fraction of filler. Carbon nanomaterials are particularly interesting; as conductive fillers they allow the enhancement of multiple properties including mechanical, electrical and thermal properties. Carbon nanotubes are very promising thanks to their diameter, D, in the nanometer range, length, L, in the micron range and their extreme aspect ratio, L/D, than can be higher than 10^6. Their main disadvantages are their high cost and their disponibility limited to low quantity. Carbon nanofibres (CNF) are cheaper and present properties close but inferior to nanotubes. They also have a larger diameter and hence a limited aspect ratio (L/D < 1000). Exfoliated graphite seems to have all the qualities (high aspect ratio, small size, extremely low price, higher disponibility) paradoxically few studies were dedicated to it.

The present work focuses on the electrical properties of carbon nanofilled epoxy composites. The performance of the three nano-carbon in terms of conductivity and transition weight fraction were investigated by means of impedance spectroscopy.

2 Experimental

2.1 Materials

Heat treated graphitized CNF (Pyrograf III PR24LHT) with diameters in the range of 60-150 nm and lengths between 30 and 100 μm, were obtained from ASI. Multiwall carbon nanotubes (MWNT) diameters in the range 13-16 nm and length between 1 and 10 μm were supplied by Bayer. Exfoliated graphite (EG) was supplied by Asbury. A low viscosity diglycidyl ether of bisphenol-A (DGEBA) epoxy resin (η ~ 0.7 Pa.s) with triethylenetetramine (TETA) hardener (Epofix, Struers) was used as matrix.

2.2 Composite preparation

The nanocomposites preparation procedure was the same for the three types of nanofillers. It involves the dispersion of the nanofiller in acetone using ultrasonication. The resin was then added and the mixture was simultaneously submitted to mechanical mixing and ultrasonic processing. The solvent was removed by heating under continuous mechanical mixing. The hardener was added last and the mixture was homogenised with a vacuum mixer.

2.3 Characterisation techniques

2.3.1 Microscopy

The nanofillers were examined using a Hitachi S4700 Field Emission Scanning Electron Microscope at 2 kV accelerating voltage to determine their morphology.

3.3.1 Impedance spectroscopy

The electrical properties were investigated using a dielectric analyzer (TA Instrument DEA 2970) in ceramic parallel plate mode. All experiments were performed at 298 K and testing frequencies ranged from 1 to 10^5 Hz. Nitrogen gas was used to provide an inert environment at a flowing rate of 500 ml/min. The sample was placed between two gold electrodes, a load (500 N) was applied and the thickness was measured with the built-in LVDT (linear voltage-displacement transducer) with a precision of 1 μm. A low amplitude sinusoidal voltage, $V_{applied}$, was applied and the current through the sample. $I_{measured}$. was measured. The AC conductivity is given by

$$\sigma_{AC} = I_{measured}/V_{applied}(\cos \delta)e/A \qquad (1)$$

with δ being the phase angle shift, e being the thickness and A the surface area of the sample.

3 Results

3.1 Morphology

The nano-carbon fillers present different shapes and sizes. CNF and MWNT have a very similar morphology with two orders of magnitude of difference in scale. They are highly entangled and randomly organized. Exfoliated graphite has a kind of accordion like structure with large galleries.

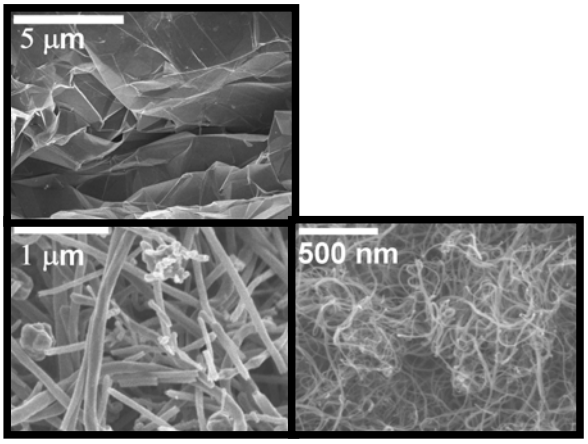

Fig. 1. Electron micrographs of the three nano-carbon fillers (MWNT, CNF, EG).

3.2 Electrical conductivity

The conductivity at 1 Hz is shown in Figure 2 as a function of the weight fraction of nanofillers for the three nano-carbon studied. It can be seen that the insulator-to-conductor transition occurred at different weight fractions depending on the type of filler. The transition occurred in this order MWNT < CNF < EG. In the case of MWNT, less than 1 wt % was sufficient to reach the transition while for CNF, a few more than 1 wt % was needed. A very high amount of EG, more than 3 wt %, led to the transition. This may be due to the low aspect ratio of the graphite nanoplates that were obtained by ultrasonic processing of the exfoliated graphite. The high aspect of MWNT and CNF allow the formation of an interconnected conductive network inside the matrix at relatively low loadings. The maximum electrical conductivity reached was in the range 10^{-7}-10^{-5} S/cm. In that case, the order was MWNT > CNF > EG. The higher conductivity was obtained with MWNT at a lower loading compared with the two other fillers. The conductivity of the epoxy/CNF nanocomposite was close to 10^{-5} S/cm for more than 4 wt %. The conductivity of the nanocomposites based on EG was two orders of magnitude lower, around 10^{-7} S/cm with more than 5 wt %. The hierarchy in maximum conductivity corresponds to that of the conductivity of the fillers. MWNT have the higher conductivity, higher than 10^{-4} S/cm along the axis as reported by the supplier. CNF have conductivity about one order of magnitude lower than MWNT. EG have an anisotropic conductivity [2], which is in the in-plane direction in the same range as CNF (10^{3} S/cm) and one order of magnitude lower in the transverse direction.

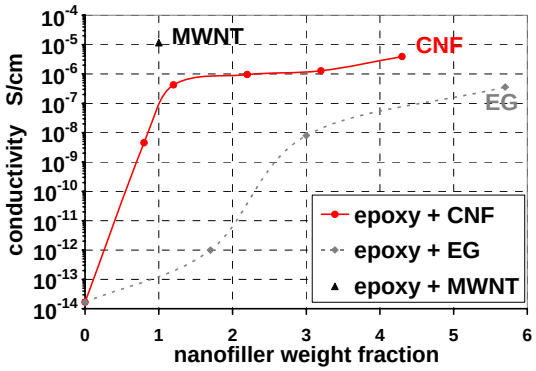

Fig. 2. Conductivity at 1 Hz as a function of weight fraction of filler for the three nano-carbon (MWNT, CNF, EG).

References

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