ELECTRICAL CONDUCTIVITY OF UNIDIRECTIONAL GLASS FIBRE REINFORCED COMPOSITE WITH CNT-MODIFIED EPOXY MATRIX

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Abstract

Fiber reinforced plastics (FRP) made of electric nonconductive glass fibres and epoxy resin modified with carbon fiber nanotubes possess a sensing function of the damage accumulations (fiber breakages, delaminations, etc.) by means of their electric resistance change during mechanical loading. Evaluation of the piezoresistive behavior of unidirectional FRP laminates are carried out by using the uniaxial tensile tests of the specimens 0 and 90º, where reinforcing fibers were directed along and transverse to the loading direction. The components of piezoresistive tensor of UD FRP are calculated by means of the gage factors of the tensioned specimens 0 and 90º experimentally determined in accordance to the relative surface electrical resistance changes, when electrical current flowed along or across the reinforcing carbon fibers. The effective electrical conductivity of an epoxy resin filled with the well-dispersed and randomly oriented carbon nanotubes (CNT) was calculated by applying the micromechanical approach based on the model of the self-consistent composite cylinder assemblage, which includes three layers of CNT, interphase and epoxy matrix. The theoretical results predicted by this model for the CNT-epoxy nanocomposite filled with volume content of CNT up to 1% agreed well enough with the experimental data available. This validates application of such micromechanical approach to estimation of the effective conductivity of CNT-polymer nanocomposites.

1. Introduction

Since carbon nanotubes (CNTs) discovery in 1991 [1], they have rapidly become a subject of considerable scientific interest of the researchers and gave rise to a large number of publications devoted to their investigations and applications. This interest is based on a unique combination of their excellent mechanical, transport, and electromechanical properties, which were confirmed experimentally and theoretically [2, 3]. The nanoscale fillers such as CNTs with at least one dimension less than 100 nm in size have significant benefits in comparison to microscale fillers owing to their high aspect ratio 10^2 - 10^5. CNTs have opened up the great opportunities for development of advanced composite materials systems that have both tailored structural and functional properties. First of all, CNTs incorporated into polymer resins results in polymer composites with dramatically improved mechanical and transport properties with potential for use in a wide range of multifunctional and high performance applications. The electrically conductive composites can be produced with high electrical conductivity at very low volume content of CNTs (often below 1%). Electrically conductive CNT-polymer composites find engineering applications in the electronic, aerospace, biomedical, automotive, and etc. industries [2].

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CNT-polymer composites are primarily composed of conductor-insulator heterogeneous material systems whose electrical conductivity can be tailored as desired. It should be noted that CNTs can significantly increase the electrical conductivity of polymers without adversely affecting other physical and mechanical properties. Owing to the change in electrical conductivity or resistivity under mechanical stress, CNT-polymer composites can be used as sensors and actuators in such potential applications as in-situ monitoring of stress distribution and active control of the composite structures [4, 5]. CNTs-based conductive polymer composites, applied as the resistance-type strain sensors provide a range of advantages over other types of strain sensors. The major advantage being that higher strain sensitivity can now be accessed. Flexible and stretchable strain sensors for use in novel applications such as large scale neuron sensor networks on engineering structures or working as human skins, as intelligent coatings or for bionic applications are now real possibilities.

The purpose of the research is an estimation of the electrical conductivity of the CNTs-epoxy nanocomposites. The micromechanical model applied for predicting the effective electrical conductivity of CNT-based nanocomposites is based on a self-consistent composite cylinders assemblage, accounting for the nanoscale effects by incorporating an interphase layer outside the CNT [6]. For validation this micromechanical model, the numerical results predicted will be compared with the experimental data available.

2. Micromechanical model for estimation of effective electrical conductivity of CNT composites

Macroscale boundary value problem concerned with determination of the effective electrical conductivity of a polymer matrix comprised of randomly oriented, well-dispersed CNTs can be solved by using methods of micromechanics of composites [6-8]. For this problem solution, we will apply the analytical approach developed [6], which is based on a multiscale modelling shown schematically in Fig. 1. Here, we briefly describe the procedure of this micromechanical approach.

On the macroscale level, CNT-polymer nanocomposite is considered as a collection of the microscale representative volume elements (RVE), consisting of the randomly oriented nanoscale RVEs. Nanoscale RVE is modelled as an assemblage of the concentric self-consistent cylinders assemblage consisting, in general, of the N layers of straight hollow carbon nanotube, surrounded interphase layer, and the outer layer $i = N$ of polymer matrix. The interphase layer intended to capture the nanoscale effects of polymer structure perturbation and/or electron hopping.

The steady state conservation of electrical charge equation at the macroscale level [9] is $\bar{J}_{i,i} = 0$, where $\bar{J}_i$ is the macroscale electric flux vector and $\bar{J}_{i,i}$ denotes the divergence of the electric flux vector in terms of the Yi coordinate system (Fig. 1). It is assumed that $\bar{J}_i$ obeys to the Ohms's law, $\bar{J}_i = \breve{\sigma}_{ij}^{\text{eff}} \bar{E}_j$ where $\bar{E}_j$ is the macroscale electrical field vector; $\breve{\sigma}_{ij}^{\text{eff}}$ is a tensor of the effective electrical conductivity of nanocomposite. Since the electrical field vector $\bar{E}_j$ can be expressed in terms of the electrical potential $\bar{\varphi}$ as $\bar{E}_j = -\bar{\varphi}_{,j}$, one can write $\breve{\sigma}_{ij}^{\text{eff}} \bar{\varphi}_{,ij} = 0$. The problem of determination of the effective electrical conductivity of CNT-polymer nanocomposite $\breve{\sigma}_{ij}^{\text{eff}}$ is solved in several stages.

Effective electrical conductivity of nanoscale RVE. The first stage of the micromechanical approach is determination of tensor of the effective electrical conductivity $\breve{\sigma}_{ij}^{\text{eff}}$ for the nanoscale RVE by using the method of concentric composite cylinders assemblage developed in [10] and generalized in [11]. This method is based on the set of homogeneous boundary conditions both for the

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multilayered composite cylinders assemblage for the nanoscale RVE and an equivalent homogeneous effective nanofiber (Fig. 1). The permissible fields of electric potential of the nanoscale RVE $\varphi^{(i)}$ and equivalent effective nanofiber $\varphi^{(\mathrm{eff})}$ should satisfy the conservation of charge equation under steady state conditions, expressed in the local coordinate system x_i, as well as to the corresponding boundary conditions.

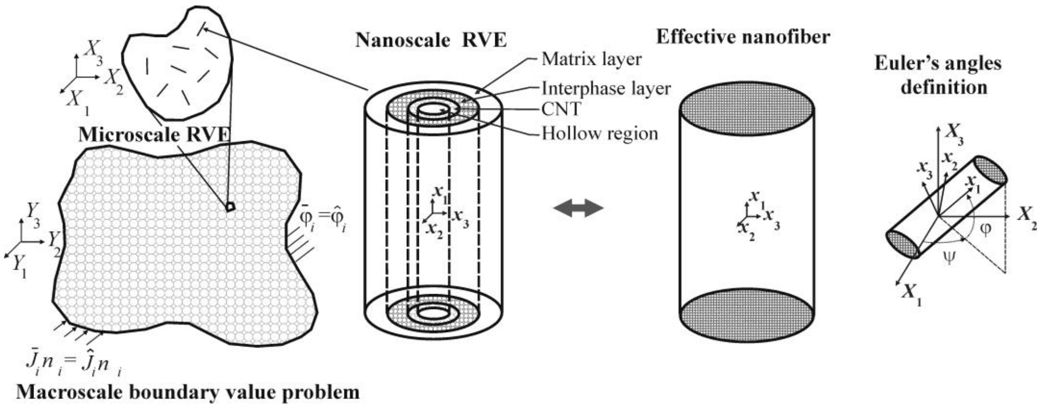

Figure 1. Scheme of a self-consistent composite cylinders model for randomly oriented, well-dispersed CNTs in polymer matrix.

The permissible fields of electric potential of the nanoscale RVE $\varphi^{(i)}$ and equivalent effective nanofiber $\varphi^{(\mathrm{eff})}$ should satisfy the conservation of charge equation under steady state conditions, expressed in the local coordinate system x_i, as well as to the corresponding boundary conditions. In the case considered, it is convenient to solve the appropriate boundary value problems in a local cylindrical coordinate system (r, z, θ) connected with the local Cartesian axes x_i ($x_1 = z$, $x_2 = r\cos\theta$, $x_3 = r\sin\theta$). The governing equation is

$$\breve{\sigma}_{rr}\frac{\partial^2\varphi}{\partial r^2}+\breve{\sigma}_{\theta\theta}\left(\frac{1}{r^2}\frac{\partial^2\varphi}{\partial\theta^2}+\frac{1}{r}\frac{\partial\varphi}{\partial r}\right)+\breve{\sigma}_{zz}\frac{\partial^2\varphi}{\partial z^2}=0$$

where $\breve{\sigma}_{rr}$, $\breve{\sigma}_{\theta\theta}$, $\breve{\sigma}_{zz}$ are the components of effective electrical conductivity of nanoscale RVE. Suggesting the transversal isotropy of the nanoscale RVE and nanofiber materials, we have that $\breve{\sigma}_{rr} = \breve{\sigma}_{\theta\theta} = \breve{\sigma}_T$ and $\breve{\sigma}_{zz} = \breve{\sigma}_L$. The equation has two permissible solutions for uniform electrical conduction in the longitudinal direction L (i.e. axis z) and in transverse direction T (i.e. axis r).

$$\breve{\sigma}_L=\frac{1}{r_N^2}\sum_{i=1}^{N}\breve{\sigma}^{(i)}\left(r_i^2-r_{i-1}^2\right),\quad \breve{\sigma}_T=\frac{1}{r_N^2E_0^2}\sum_{i=1}^{N}\breve{\sigma}^{(i)}\left[\left(A_1^{(i)}\right)^2\left(r_i^2-r_{i-1}^2\right)-\left(A_2^{(i)}\right)^2\left(\frac{1}{r_i^2}-\frac{1}{r_{i-1}^2}\right)\right]$$

Flux concentration tensor approximation. The procedure of determination of the effective properties of a multi-phase composite by means of any micromechanical approach requires the determination of appropriate concentration tensor components. According to Hill's conception [12], this tensor transforms the averaged quantity (e.g. stress, strain, electric flux, etc.) into a phase averaged quantity and characterizes a degree of interactions between the inclusions in the multi-phase composite. Some of the micromechanical methods developed (e.g., Mori-Tanaka method [13]) apply the concentration tensor. However, in the case of the multi-layered self-consistent composite cylinders

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method, the required components of electric flux concentration tensor $B_{jk}^{(\text{local})}$, expressed in the local coordinate system of the nanoscale RVE, can be calculated by using its immediate determination [14]

$$\left\langle J_j^{(N-1)} \right\rangle = B_{jk}^{(\text{local})} \left\langle J_j^{(N)} \right\rangle$$

where $\left\langle J_j^{(N)} \right\rangle$ refers to the electric flux averaged over the volume of the entire composite cylinders assemblage (i.e., over all N layers: $i = 1,\ 2, \ldots,\ N$), while $\left\langle J_j^{(N-1)} \right\rangle$ refers to the volume average of the flux over just the CNT and interphase layers of the assemblage (i.e., over N-1 layers: $i = 1,\ 2, \ldots,\ N-1$). Finally, the volume averaged electric fluxes can be calculated by the formulas [14]

$$\left\langle J_i^{(N-1)} \right\rangle = \frac{1}{\pi r_{N-1}^2 L} \sum_{j=1}^{N-1} \int_{-L/2}^{L/2} \int_0^{2\pi} \int_{r_{j-1}}^{r_j} J_i^{(j)} r dr d\theta dz, \quad \left\langle J_i^{(N)} \right\rangle = \frac{1}{\pi r_N^2 L} \sum_{j=1}^{N} \int_{-L/2}^{L/2} \int_0^{2\pi} \int_{r_{j-1}}^{r_j} J_i^{(j)} r dr d\theta dz$$

Effective electrical conductivity of nanocomposite. One of the key assumptions accepted for the microscale RVE of the nanocomposite studied is that CNTs are well-dispersed and randomly oriented in the polymer matrix. This allows us consider the nanocomposite at microscale level as a two-phase microscale RVE consisting of similar solid equivalent nanofillers with random orientations in the polymer matrix. Orientation of the each nanofiller in the microscale RVE is determined by two Euler's angles ψ and φ (see Fig. 1). Considering that the microscale RVE is able to statistically represent the overall effective electrical conductivity of the nanocomposite, we can apply method of orientational averaging [15] for determination of the effective electrical conductivity of the nanocomposite. According to this method, tensor of the effective electrical conductivity of the nanocomposite $\breve{\sigma}_{ij}^{(\text{eff})}$ can be calculated as

$$\breve{\sigma}_{ij}^{(\text{eff})} = \breve{\sigma}_{ij}^{(m)} + \frac{v_f}{4\pi} \int_0^{2\pi} \int_0^{\pi} \left[\breve{\sigma}_{ik}^{(\text{global})}(\varphi,\psi) - \breve{\sigma}_{ik}^{(m)} \right] A_{kj}^{(\text{global})}(\varphi,\psi) \sin\varphi \, d\varphi \, d\psi$$

where $\breve{\sigma}_{ij}^{(m)}$ is tensor of the electrical conductivity of polymer matrix $\breve{\sigma}_{ij} = \breve{\sigma}_m,\ i = j$, $\breve{\sigma}_{ij} = 0,\ i \neq j$; $\breve{\sigma}_{ik}^{(\text{global})}(\varphi,\psi)$ and $A_{kj}^{(\text{global})}(\varphi,\psi)$ are the electrical field conductivity and concentration tensors, respectively, expressed in the global coordinate system X_i; v_f is volume content of CNTs in the nanoscale RVE.

3. Experimental study

Nanocomposites used for the electrical conductivity experimental studying in LU PMI were made from the commercially available epoxy resins filled with different types of CNTs.

The first nanocomposite consists of DGEBA-based epoxy resin (L135i) with an amine hardener (H137i) (supplied by Momentive Specialty Chemicals, Stuttgart, Germany) and multi-wall carbon nanotubes Baytubes C150P (Bayer, Germany). This resin system is characterized by low viscosity (~ 250 mPa·s) and widely applied for fibre-reinforced composites produced by an infusion technology. According to the producer's data sheet [16], electrical conductivity of the Baytubes C150P is more than 10^6 S/m, length is 1-10 μm, inner and outer radii are 2 and 5-10 nm, respectively. The DGEBA/Baytubes C150P nanocomposite with different weight content of CNTs (0.05, 0.3, 0.5, and 1% wt.) was prepared by via a shear-mixing technique.

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The second nanocomposite consists of mono-component thermosetting epoxy resin RTM6 (HexFlow®) and multi-wall carbon nanotubes N7000 (Nanocyl S. A., Belgium), which were used without any purification treatment. This epoxy system was specially developed for the aerospace industries and applied in the resin transfer moulding technology. This epoxy resin is characterized by high glass transition temperature (200 ºC) and low density (~50 mPa·s) with the temperature range of 100-120 ºC. According to the producer's data sheet [17], carbon nanotubes N7000 have the following average characteristics: length of 10 μm, diameter of 9.5 nm, and aspect ratio of 500. This nanocomposite (RTM6/NC7000) was prepared also with different weight content of CNTs (0.03, 0.05, 0.15, and 0.30% wt.) by using a high-performance disperser.

The plates of the DGEBA/Baytubes C150P and RTM6/NC7000 nanocomposites fabricated were cut on the flat specimens (110×10×2.2 mm). For electrical conductivity measurement, the electrodes made of a silver conductive paste were formed on the specimen surfaces. The specimens' electric resistance *R* was measured by the two-probe method, based on Ohm's law. The averaged values of specific resistance $\breve{\rho}$ of the nanocomposites calculated from at least three tested specimens are presented in the Table 1.

Table 1. Values of specific resistance of nanocomposites at different weight content of CNTs.

v_f (% wt.)	$\breve{\rho}$ (KΩ·m)	
	RTM6/NC7000	DGEBA/Baytubes C150P
0.03	155 ± 73	–
0.05	1.71 ± 0.08	12.5 ± 1.2
0.15	0.033 ± 0.002	–
0.30	0.0083 ± 0.0003	1.0 ± 0.04
0.50	–	0.3 ± 0.01
1.00	–	0.1 ± 0.005

4. Calculational and experimental results discussion

The micromechanical approach described was applied for calculation of the effective electrical conductivity of the nanocomposite containing the CNTs well-dispersed and randomly oriented in an epoxy resin. The nanoscale RVE of the nanocomposite consists of three layers: CNT (*i* = 1), interphase (*i* = 2) and matrix (*i* = 3) (see Fig. 1). Thus, the micromechanical model includes the following parameters: outer radius r_1 and thickness $t_1 = r_1 - r_0$ of CNT, thickness of the interphase $t_2 = r_2 - r_1$ and matrix $t_3 = r_3 - r_2$ layers, electrical conductivity of the layers $\breve{\sigma}_1, \breve{\sigma}_2, \breve{\sigma}_3$, and volume content of CNTs in the nanocomposite v_f. The outer radius r_3 of the matrix layer equalled to the outer radius of the composite cylinder assemblage r_{out} is determined $r_3 = r_1 / \sqrt{v_f}$.

The results of numerical modelling will undoubtedly depend on the values of the micromechanical model parameters selected. It should be noted that experimental determination of some parameters of the micromechanical model, such as thickness and conductivity of the interfacial layer, thickness and inner diameter of CNTs, is associated with experimental difficulties. There is uncertainty about the ranges of their changes in the scientific papers published. Therefore, the initial values for these parameters were taken as in [7]: $\breve{\sigma}_2 = \breve{\sigma}_{\text{int}}$ = 10-6 S/m, r_1 = 1.0 nm, t_1 = 0.34 nm, t_2 = 2.5 nm. Moreover, $\breve{\sigma}_3 = \breve{\sigma}_m$ = $9 \cdot 10^{-9}$ S/m for DGEBA-based epoxy resin as in [8].

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The reliability of the effective electrical conductivity of the nanocomposites calculated by means of the micromechanical approach was initially verified by comparison with the experimental data available. For this purpose, numerical calculations were carried out with different values of the electrical conductivity of CNTs: $\breve{\sigma}_1 = \breve{\sigma}_{CNT} = 10^7$ (for NC7000), $1.85{\cdot}10^5$, $5{\cdot}10^4$, and 10^4 S/m taken from [17-20], respectively. The results shown in Fig. 2 demonstrate a fairly good agreement between the theoretical prognoses and the experimental data presented in [18–20], as well as for the RTM6/NC7000 nanocomposite (see Table 1).

However, the values of effective electrical conductivity calculated for the DGEBA/Baytubes C150P nanocomposite were significantly greater then experimental ones (see Table 1). It seems, that the main reason is calculation with the overestimated value $\breve{\sigma}_1 = 10^6$ S/m chosen according with the producer's data sheet [16]. The results of experimental investigation of electrical conductivity of the nanocomposites filled with different multi-wall CNTs [21, 22] confirm this assumption. The measured values of electrical conductivity for the nanocomposites filled with Baytubes C150P were the lowest ones.

Thus, we can conclude that good enough agreement observed between the analytical and experimental results validates the application of the micromechanical model based on the concept of self-consistent composite cylinder assemblage for estimation of the effective electrical conductivity of the polymer resins filled with well-dispersed and randomly oriented CNTs.

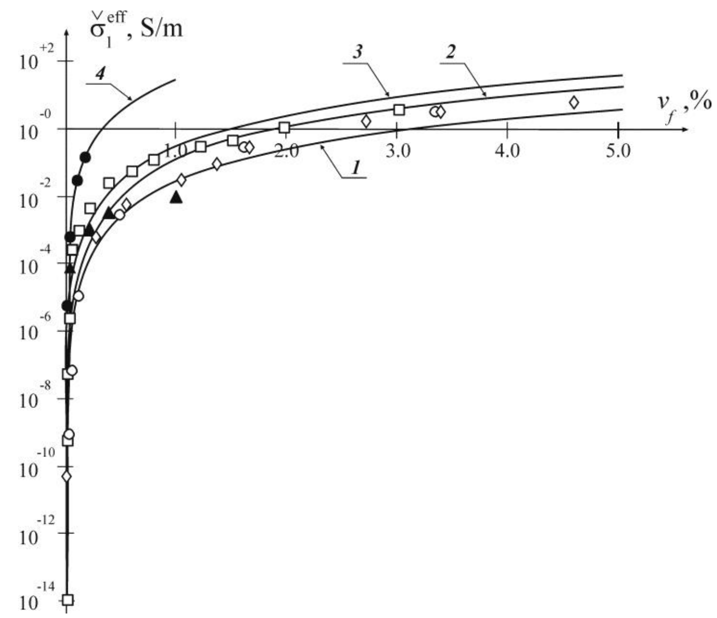

Figure 2. Dependence of effective electrical conductivity of CNT-epoxy nanocomposite $\breve{\sigma}_1^{eff}$ on volume content of CNTs v_f . Theoretical prediction (curves 1–4) and experimental data: □[18]; ○[19], ◇[20] and ▲, ● [LU PMI].

The RTM6/NC7000 nanocomposite has the lowest electrical percolation threshold, while the DGEBA/Baytubes C150P nanocomposite requires the highest amount of nanotubes to achieve percolation (see Fig. 2). The same results were obtained in [21, 22] for the nanocomposites filled with different carbon nanotubes. Such different behaviour of the DGEBA/Baytubes C150 and RTM6/NC7000 nanocomposites is explained by a much higher electrical conductivity of NC7000 nanotubes, greater length and aspect ratio in comparison with Baytubes C150P21. Moreover, the paper [21] concludes that the infiltration process of polymer resin chains into Baytubes C150P nanotubes is more difficult, which leads to a lower level of dispersion, and as a result requires their higher amount to reach the electrical percolation threshold. The experimental values of the electrical percolation threshold, lower then those calculated according to geometrical continuum percolation theory [21], can be explained by such nanoscale effects as hopping [6, 23–25] or tunnelling [25–27] for electron transport, which do not require direct contact between the carbon nanotubes. However, the question about the true mechanism of providing such a low percolation threshold is debatable until now. There are different opinions on a fact of a low experimental electrical percolation threshold of the nanocomposites may be associated with both the electron hopping and the formation of conductive networks within the polymer matrix. Nevertheless, this fact characterizes their extreme sensitivity and opens a way for the development of advanced sensors on their base [5, 28].

The effects of material and geometry parameters (electrical conductivity and thickness of CNTS, thickness of interphase layer) of the micromechanical model on the effective electrical conductivity of CNT-epoxy composite were studied by means of the parametric analysis. Obtained numerical results are presented in [29].

5. Conclusions

The micromechanical model based on the self-consistent composite cylinder assemblage is applied for calculating the effective electrical conductivity of the epoxy resin filled with the well-dispersed and randomly oriented carbon nanotubes. The results predicted by this model agree enough well with the experimental data available, and this validates application of this micromechanical approach for estimating the effective electrical conductivity on CNT-polymer nanocomposites. The results of parametric analysis showed that the effective electric conductivity of CNT-polymer nanocomposite can be enhanced by increasing the electric conductivity of CNTs, as well as the interphase layer thickness, which is responsible on the so-called electron hopping mechanism in the CNT-polymer nanocomposites. The low percolation threshold decreases also with increasing the values of these parameters of the micromechanical model.

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