ELECTRO-MECHANICAL TRANSDUCER BASED ON CARBON NANOTUBE NETWORK/POLYSTYRENE LAMINATE FOR DEFORMATION DETECTION

SLOBODIAN Petr[1c], KOVAR Michal[1b], OLEJNIK Robert[1c], MATYAS Jiri[1d]

[1]Centre of Polymer Systems, University Institute, Tomas Bata University, Trida T. Bati 5678, 760 01 Zlin, Czech Republic

[a]slobodian@cps.utb.cz, [b]kovarmichal@seznam.cz, [c]olejnik@cps.utb.cz, [d]matyas@cps.utb.cz

Abstract

A new type of polystyrene (PS)/carbon nanotube (CNT) network laminate is introduced as an electrically conductive composite material; with favorable properties as electro-mechanical signal transducer capable to detect applied mechanical strain. In course of its fabrication a non-woven polystyrene membrane made by electro spinning was used as filtering mesh for CNT aqueous dispersion. Produced semi-product like filtering membrane with entrapped carbon nanotubes was stuck using solvent of PS on polystyrene test specimen. The electrical resistance of final laminate is sensitive to tensile strain when elongation leads to increase of macroscopic electrical resistance. Test specimens were then tested in the course of monotonic strain growth and also when loading/unloading cycles were imposed. Changes in resistance were found to be reversible, reproducible and deformation can by monitored in real time. Finally, sensitivity to strain can be quantified by means of a gauge factor, GF, which defines sensitivity of strain gauge as a relative resistance change divided by the applied strain. Measured GF for PS/CNT laminates reaches relatively high values, compared with ones of commonly used metallic strain gauges, serving for values of around 13 and applied tensile deformation in range 0.1-0.6 %. These experimental results are really promising serving for real practical application of this principle in course of polymeric based strain gauges or as an integrated PS/CNT units into polystyrene based constructions for their so called “health monitoring”.

Keywords: Carbon nanotubes networks, CNT, polystyrene, strain gauge, electro-mechanical transducer

1. INTRODUCTION

It was found that interconnected carbon nanotube (CNT) structures like carbon nanotube networks, so called Buckypapers, are capable to detect their deformation by change of macroscopic electrical resistance [1-3]. In the case of the CNT entangled network, the resistance response to deformation is governed by nanotube interactions. The decisive role is played by local resistance in nanotube contacts and the straightening and buckling of nanotubes what change the number of contacts between nanotubes, rather than the change of intrinsic piezoresistive properties of individual CNT [2]. Polymeric strain sensitive sensors can by manufactured when such CNT active element are incorporated into a polymer matrix or may optionally serve as a structural element for health monitoring of polymeric products or structures made of polymeric materials. The published data show that the electrical response to strain or stress is sufficient and the sensing can be performed in real time [1-7]. Moreover, the deformation process is reversible although some irreversibility is also measured as a hysteresis loop in cyclic loading tests, or as a residual resistance which remains after specimen unloading [1-7].

In this paper we prepared polymer nanocomposite which consists of multi-walled carbon nanotube (MWCNT) entangled network and polystyrene matrix by innovative procedure when the non-woven polyurethane filtering membrane and the carbon nanotube filtration cake are integrated by compression moulding. Prepared CNT/PS composites are strain sensitive by their change of macroscopic electrical resistance. Electro-mechanical behavior was studied under elongation and elongation/relaxation cycles.

2. EXPERIMENTAL

Polystyrene (PS) non-woven membrane for filtration of nanotube dispersion was prepared by electrospinning from a commercial polymer (Krasten 137, Kaucuk-Unipetrol Group). The polymer was dissolved in a mixture of methyl isobutyl ketone and dimethylformamide with the volume ratio 3:1 and PS concentration 15 wt. %. PS nanofiber layer was manufactured using the laboratory apparatus SPIN-LAB (SPUR a.s., Czech Republic) equipped with a electrospinnig needles electrode and a steel planar collecting electrode. Electrospinning was carried out under the following conditions: electric voltage 75 kV temperature 20-25°C, relative humidity 25-35 %. To produce final PS non-woven filters, the prepared nanofiber porous layer (thickness of about 1 mm) was subjected to hot pressing under 0.6 MPa and temperature 80°C.

Purified multi-walled carbon nanotubes (MWCNT) produced by chemical vapor deposition of acetylene were supplied by Sun Nanotech Co. Ltd., China. According to the supplier, the nanotube diameter is 10-30 nm, length 1-10 μm, with a purity of ~90% and (volume) a resistivity of 0.12 Ωcm. Further details on the nanotubes were obtained by means of the transmission electron microscopy (TEM) analysis presented in our paper [8]. From the corresponding micrographs the diameter of individual nanotubes was determined to be between 10 and 60 nm, their length from tenths of micron up to 3 μm. The maximum aspect ratio of the measured nanotubes is thus about 300. The multi-wall consists of about 15-35 rolled layers of graphene.

In order to prepare entangled CNT network on the supporting PS filter, a vacuum-filtration method was used. The nanotubes (0.3 wt.%) were dispersed in water with dissolved sodium dodecyl sulfate (SDS) and 1-pentanol with concentrations 0.1M and 0.14M, respectively, by sonication with UZ Sonopuls HD 2070 kit. Consequently, an aqueous solution of NaOH was added to adjust the pH at value of 10. The thickness of the non-woven PS filter was typically 0.5-0.8 mm and the thickness of MWNT entangled network, according of the amount of dispersion filtered, was from 0.02 to 0.26 mm. Finally, carbon nanotube network/polystyrene laminate for the tensile extension/resistance tests was prepared from CNT network on the supporting PS filter and PS dog-bone shaped specimen made according to the standard EN ISO 3167. At the first step, the CNT network on the supporting PS filter was compression moulded at processing temperature 190 °C (well above the glass transition temperature (T_g) of used PS). At this process, fibres of PS filter are melted and filter is transformed into PS thin film with interlocked carbon nanotubes network. At the second step MWNT/PS thin film composite stripe cut (length 30 mm and width 5 mm) is fixed on PS dog-bone shaped specimen using 20 wt. % solution of PS in butanone. Two electrical contacts were fixed to the stripe by silver colloid electro-conductive paste (Dotite D-550, SPI Supplies) and the electrical resistance was measured lengthwise during 7 consecutive tensile loading/unloading cycles. Resistance was measured by the two-point technique using a multimeter (Sefram 7338).

3. RESULTS AND DISCUSSION

The upper surface of the PS non-woven filtrating membrane produced by SEM is presented in Fig. 1 a). PS fibres are straight with smooth surface and submicron sizes with the average diameter of 0.6± 0.3 μm. The pores between them have an average size of about 0.5 μm and its porosity is about 0.9. TEM micrograph in Fig. 1 b) gives a detail view of used nanotubes which are not very good quality with a large number of defects in its structure. On the other hand they are relatively cheap.

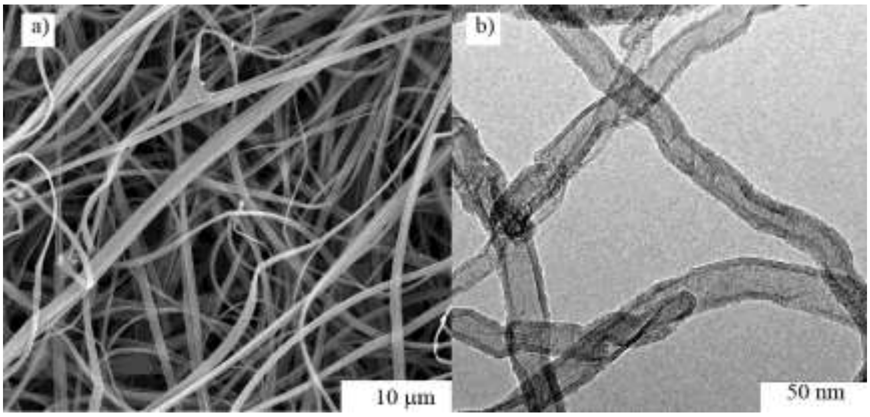

Fig. 1 Part a) SEM analyses of PS non-woven filtrating membrane prepared by technology of electrospinning (Vega LMU, produced by Tescan Ltd.). Part b) TEM analyses of used multi-walled carbon nanotubes (MWCNT) (JEOL JEM 2010).

The pores of PS non-woven filtrating membrane allow partial infiltration of MWCNT into the filter at the beginning of filtration. It can be documented by SEM analysis of profile view of cut thought PS filter/MWCNT structure where arrow indicates filtration direction, see Fig. 2 a). Then the pores are filled with nanotubes, the filter cake (pure nanotube entangled network) is formed above the filter surface. SEM analyses of upper surface of the MWCNT network is presented in Fig. 2 b). MWCNT network is a porous (porosity ~ 0.67) and electrically conductive structure (conductivity ~ 11.9 S/cm) created by entangled nanotubes with electrical conductive junctions between them. The electrical conductivity of the MWCNT network is predominantly determined by the contact resistance in junctions of crossing nanotubes, rather than by resistance of MWCNTs itself. The CNT network on the supporting PS filter was compression moulded when PS non-woven filtrating membrane was melted forming PS thin film with interlocked carbon nanotubes network, see Fig. 2 c).

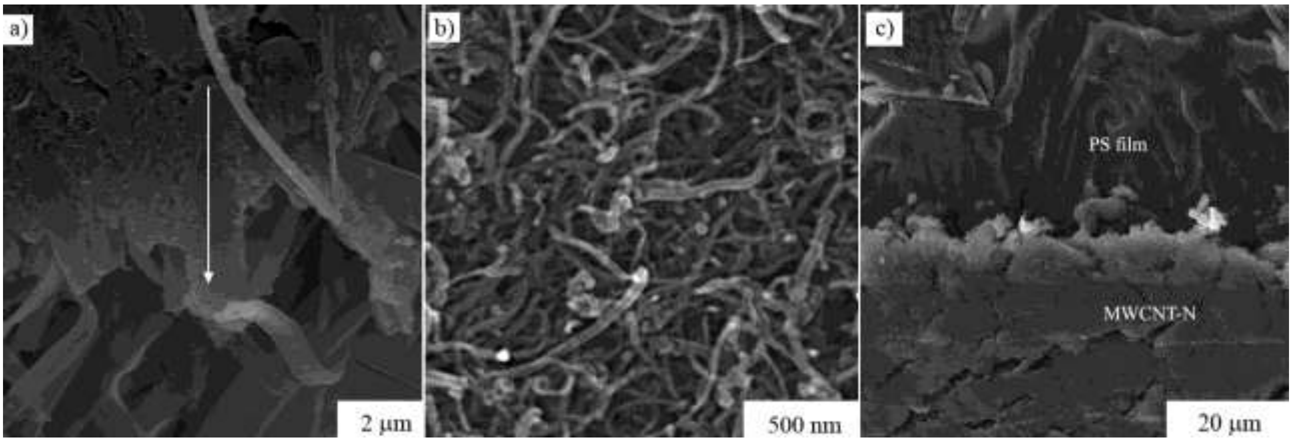

Fig. 2 Part a) SEM analyses of profile view of cut thought PS filter/MWCNT structure where arrow indicates filtration direction. Part b) SEM image of the surface of entangled MWCNT network. Part c) SEM analyses demonstrating the transformation of PS non-woven filtrating membrane into form of PS thin film with interlocked carbon nanotubes network.

Finally, MWNT/PS thin film composite stripe cut was fixed on PS dog-bone shaped specimen using 20 wt. % solution of PS in butanone, Fig. 3. This electro-mechanical transducer was exposed to strain in apparatus applying creep tensile load. Strain/resistance values were counted after 20 s of loading/unloading cycles.

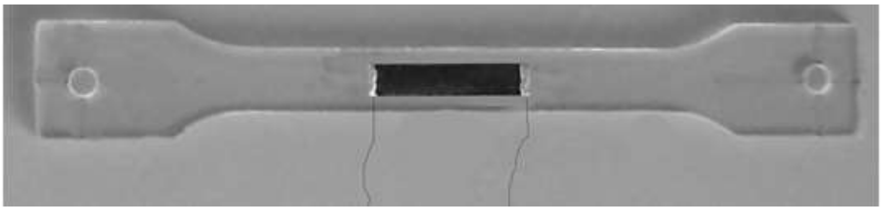

Fig. 3 Dog bone shape of PS specimens with the fixed stripe of PS/MWNT thin film composite.

Strain-electric experimental data are presented with the help of the percentage of relative resistance change:

$$\frac{\Delta R}{R_0} = \frac{R - R_0}{R_0} \qquad (1)$$

where R_0 is the electrical resistance of the measured sample before the first elongation, and R is the resistance while elongating. And strain:

$$\varepsilon = \frac{L - L_0}{L_0} \qquad (2)$$

where L_0 represents initial length of PS specimen.

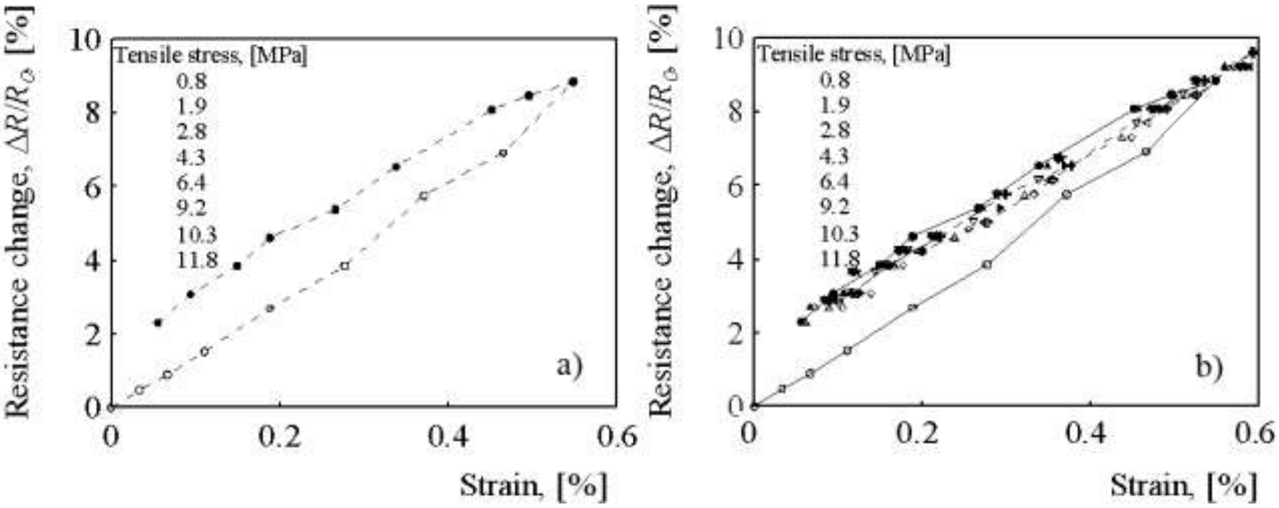

Fig. 4 Part a) Resistance change vs. strain for MWCNT/PS composite in loading/unloading. Part b) Seven loading/unloading cycles MWCNT/PS composite.

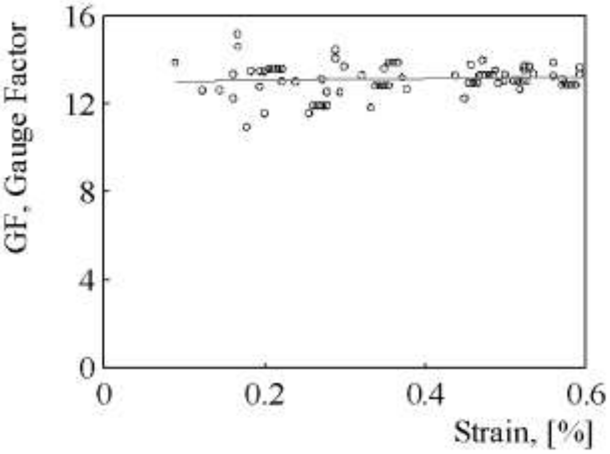

Fig. 5 Dependence of the sensitivity of strain gauge, Gauge factor, GF, on applied strain.

Examples of practical measurement are presented in Fig. 5 parts a-b). It represents resistance change on applied strain for MWCNT/PS composite during loading/unloading in one cycle and during seven consecutive loading/unloading cycles. The resistance of the MWCNT/PS gauge increase by deformation and decrease during unloading. The resistance change for maximal elongation 0.55 % was measured to be 8.85 %. Process is reversible although some irreversibility as a hysteresis loop and residual resistance after unloading can be observed. Stabilized cyclic regime is reached after the first loading/unloading cycle, see Fig. 5 part b. The sensitivity of strain gauge can be defined as the relative resistance change divided by the applied strain by means of a gauge factor (GF):

$$GF = \frac{\Delta R / R_0}{\varepsilon} \tag{3}$$

GF presented as strain dependence in Fig. 5 reaches values around 13 and is independent on applied strain in range 0.1-0.6 %. This value is relatively high comparing with conventional materials usually used for construction of strain gauges such as metal foil strain gauges (GF ~ 2-5) or Copper (GF ~ 2.5).

4. CONCLUSION

The MWCNT/PS strain sensitive nanocomposite was prepared by the innovative procedure when the non-woven polystyrene filtering membrane and the carbon nanotube filtration cake were bonded by the compression molding finally sticking on PS dog-bone shaped test specimen by PS based glue. The SEM observation indicates the penetration of carbon nanotubes into the nonwoven polystyrene filtering membrane and their tight bonding after the compression molding. The mechanical testing reveals that straining of the network leads to change of its macroscopic electrical resistance and MWCNT/PS strain sensitive nanocomposite is relatively sensitive to strain and changes are reversible. Thus, the testing indicates a good potentiality of the composite composed of electrically conductive entangled carbon nanotube network embedded in a polystyrene matrix as elements for strain detection or as an active part of sensing structural composites.

ACKNOWLEDGEMENTS

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic – Program NPU I (LO1504). This project was also supported by the internal grant of TBU in Zlin No. IGA/CPS/2015/001 funded from the resources of the Specific University Research.

REFERENCES

[1] ZHANG Z.C., WEI H.Q., LIU Y.J., LENG J.S. Self-sensing properties of smart composite based on embedded buckypaper layer. Structural Health Monitoring-An International Journal, Vol. 14, No. 2, 2015, pp. 127-136.

[2] REIN M. D., BREUER O., WAGNER H. D. Sensors and sensitivity: Carbon nanotube buckypaper films as strain sensing devices. Composites Science and Technology, Vol. 71, No. 3, 2011, pp. 373-381.

[3] DE LA VEGA A., KINLOCH I.A., YOUNG R.J., BAUHOFER W. Simultaneous global and local strain sensing in SWCNT-epoxy composites by Raman and impedance spectroscopy. Composites Science and Technology, Vol. 71, No. 2, 2011; pp.

[4] THOSTERSON E.T., CHOU T.W. Real-time in situ sensing of damage evolution in advanced fiber composites using carbon nanotube networks. Nanotechnology, Vol. 19, No. 21, 2008, article no. 215713.

[5] PHAM G.T., PARK Y.B., LIANG Z., ZHANG C., WANG B. Processing and modeling of conductive thermoplastic/carbon nanotube films for strain sensing. Composites part B-engineering, Vol. 39, No. 1, 2008, pp. 209-216.

[6] SLOBODIAN P., RIHA P., OLEJNIK R., CVELBAR U., SAHA P. Enhancing effect of KMnO4 oxidation of carbon nanotubes network embedded in elastic polyurethane on overall electro-mechanical properties of composite. Composites Science and Technology, Vol. 81, 2013, pp. 54-60.

[7] SLOBODIAN P., RIHA P., SAHA P. A highly-deformable composite composed of an entangled network of electrically-conductive carbon-nanotubes embedded in elastic polyurethane. Carbon, Vol. 50, No. 10, 2012, 3446-3453.

[8] SLOBODIAN P., RIHA P., LENGALOVA A., SAHA P. Journal of Materials Science, Vol. 46, No. 9, 2011, pp. 3186-3190.