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High elastic polyurethane/carbon nanotube composite laminate for structure health monitoring by gain shifting of antenna sensing element

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Abstract. The composite of carbon nanotubes and polyurethane (PU) was prepared by simple filtration technique. The PU nonwoven filtration membrane was prepared by electrospinning. A layer of carbon nanotubes was prepared by vacuum filtration on the surface of PU membrane. The resulting composite was subsequently placed on highly elastic polyurethane substrate. The contribution shows an efficient method of preparing the sensing element for monitoring the state of strain of loaded structures by using highly elastic polyurethane / carbon nanotubes composite. This sensor has been involved as passive antenna with stable resonance frequency of 650 MHz. When it is get deformed in the range from 0 to 3.5% the sensor gain was changing from -39 dB to - 19.45 dB. But if it is get deformed by 15% and again measured strain from 0 to 3.5%, sensor gain was changing from -33 dB to -12.3 dB, which clearly indicates the damage of structure.

1. Introduction

Carbon nanotubes network in the form of very thin layer was prepared by simple filtration method. For filtration was used non-woven structured polyurethane membrane prepared by electrospinnig method. The network was placed on polymer substrate by hot pressing method and the composite laminate was made [1]. The carbon nanotubes composite laminate has the new additional properties such as the strength, the electrical and thermal conductivity.
PU/CNT network as a strain sensor for structure health monitoring has potential advantage in that the networks have high sensitivity to local distributions of stress and strain as well as the ability to sense stresses and strains in different directions [2]. PU/CNT network as a strain sensor can be used for monitoring of crack formation or propagation in the monitored material or construction [3]. There are a lot of technique how monitored and analyzed the signal from sensing element. The sensing element signal can be measured as a change of resistivity during applying strain or change of voltage when we use Wheatstone bridge and many others.

The idea of this article is make a fusion between very elastic polymer material in the form of layered composite and carbon nanotubes as an electrical conductive material in the form of network [4]. The composite was used in the form of micro-strip antenna sensing element. The sensing element combined very elastic nonconductive polymer on one side, with stress/strain sensitive conductive layer on other side.

In this study, the stimulation effect of preloading on the gain changing properties of thermoplastic polyester-based polyurethane (TPU) laminate with MWCNT-Ns is investigated under elongation and elongation/relaxation cycles and compared with corresponding properties of CNT-N/TPU laminates without preloading. CNT-N/TPU laminates strain sensing element is able to sensitive react a clearly shows the change of gain after preloading which simulate the damage of construction.

2. Experimental

Purified MWCNT produced by chemical vapor deposition (CVD) 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, purity >90% and (volume) resistivity 0.12 cm-1. Further details on the nanotubes and the results of TEM analysis can be found in our paper [8] 2.65 g of MWCNT, sodium dodecyl sulfate (SDS) and 1-pentanol. Consequently, NaOH aqueous solution was added to adjust pH to the value of 10. The final nanotube concentration in the suspension was 0.49 wt.%, concentration of SDS and 1-pentanol 0.1M and 0.14M, respectively. The suspension was sonicated in Dr. Hielscher GmbH apparatus (ultrasonic horn S7, amplitude 88 µm, power density 300 W/cm^2, frequency 24 kHz) for 15 minutes and the temperature of ca 40°C.

The polyurethane granules were dissolved in a mixture of dimethyl formamide/methyl isobutyl ketone (Penta Chemikalie, Czech Republic) with volume ratio 3:1. The polymer weight concentration was adjusted to 16 % (w/v) and the mixture electric conductivity to 30 s/cm by adding NaCl in order to optimize the process. The total number of nozzles was 18, the length of nozzles 30 mm, the distance between nozzles 20 mm, the nozzle internal diameter 1.2 mm and the outer diameter 2.2 mm. The electric voltage was set to 75 kV (Matsusada DC power supply), the temperature 21 ± 2 °C, the relative humidity 35 ± 2.5%.

Trough above prepared membrane was filtrated carbon nanotubes dispersion. The dispersion was filtrated by using standard water vacuum filtration apparatus. Prepared filtration cake was rinsed by 65 °C hot water and then by methanol. Filtration membrane with carbon nanotubes layer was placed between two filtration paper and leave without disturbing for 24 hours to reach dry PU/CNT composite structure. From PU/CNT layer was cut sample with dimension 10 x 50 mm. The simple was place on PU base material and put together by molding machine at 175 °C. The PU/CNT composite laminate in the form of strip was mounted in the creep test apparatus. Creep test apparatus was used for extension/relaxation cyclic testing.

The speciment was equipped with elastic wire. This wire was connected with ground plane. The ground plane was equipped with coaxial cable which is connected with spectral analyzer. The sample is used as an antenna. The measurements of the antenna were performed using the R&S FSH3 spectrum analyzer (the model with a tracking generator and without a preamplifier) and FSH-Z2 bridge capable of measuring within a bandwidth of 100 kHz to 3 GHz. The frequency range was set from 100 kHz to 1.5 GHz.

3. Results and discussion

The transmission electron microscopy (TEM) micrograph in Fig. 1 a) gives a detail view of used nanotubes. There is clearly visible multi wall structure of carbon nanotubes. Individual nanotubes are entrapped by non-woven filtration membrane during vacuum filtration of MWCNT aqueous dispersion. The nanotubes are infiltrated into membrane pores and the filtering cake is made. [7]. The scanning electron microscope (SEM) analysis of mentioned PU non-woven filtering membrane is presented in Fig. 1 b). SEM micrograph in Fig. 1 c) shows the upper surface of MWCNT network. MWCNT network is a porous and electrically conductive structure 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 nanotubes are much shorter than the dimension of straining sample and inter-tube contacts act as parallel resistors between highly conductive MWCNT segments. Fig. 1 d) shows detail view of created carbon nanotubes entangled network. The final step of the laminate preparation is the melt welding of PU filter and MWCNT resulting about 2 mm thick composite plates. Electromechanical sensitivity of the prepared laminate was tested using dog-bone shape specimens cut out from two-layer laminate, see Fig. 1 e).

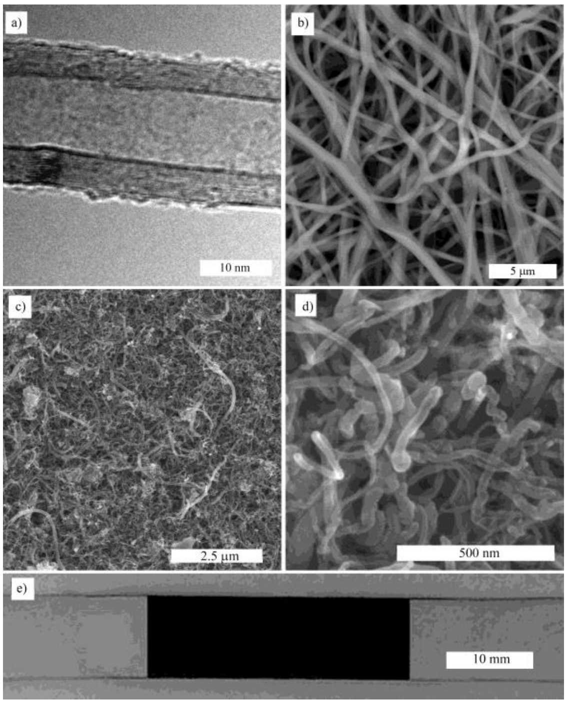

Fig. 1 a) TEM micrograph of multi wall carbon nanotube, b) SEM micrograph of polyurethane non-woven filtering membrane, c) SEM image of the surface of entangled MWCNT network, d) Detail of entangled MWCNT network e) Image of PU strip shaped specimen, for the stress/stain test for gain shift measurement with the fixed stripe of MWCNT/PU laminate (black).

The mechanical/electromagnetical behavior of carbon nanotubes network/polyurethane laminates in the form without preloading is presented in Fig. 2a. The data represents ten extension/relaxation cycles. The cycle deformation gradually increases in each 5 minutes cycle up to maximal strain value approximately 3.5 %. The preload stimulated form are presented in the following Fig. 2 b. The data represents ten extension/relaxation cycles. The cycle deformation gradually increases in each 5 minutes cycle up to maximal strain value approximately 3.25 %. The stimulated laminate was prepared by preload elongation 15%. The mechanical/electromagnetical testing was then performed after 20 hours of specimen relaxation. Both speciments form (without and preloaded) was simultaneously tested as an antenna and also extension/relaxation cycles. The gain of this antenna was measured. The strain is defined $\varepsilon = \Delta L/L_0$, where ΔL represents the change in specimen length and L_0 is the initial length before the first elongation.

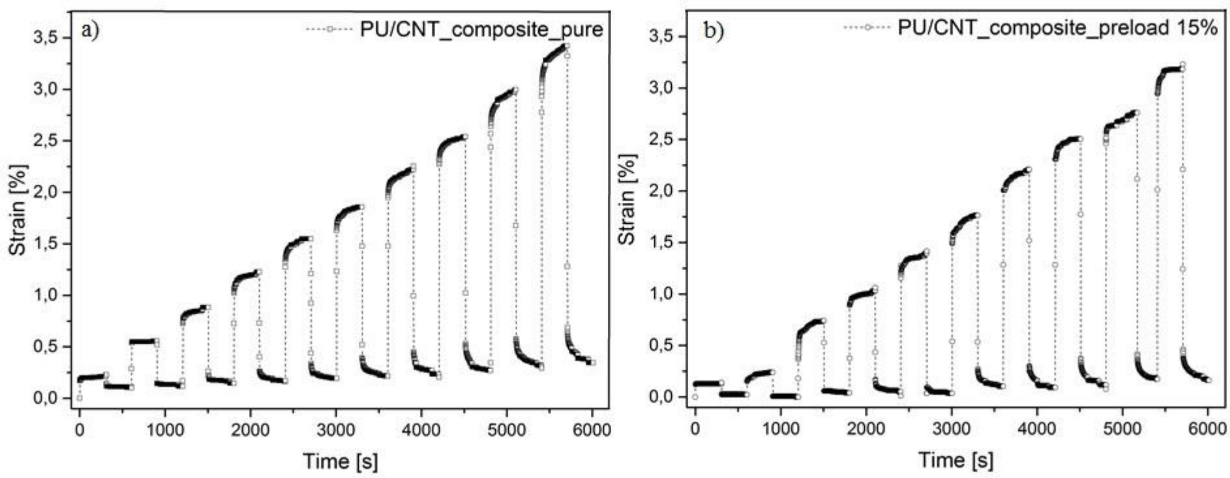

Fig. 2 Mechanical response to applied strain, ε, for MWCNT/PU laminate composite sample. a) No preload specimen, b) preload 15 %. The tensile strain step increases in a ten consecutive extension/relaxation cycles.

In the course of strain cycles, some irreversible changes in strain and the relative resistance are observed due to possible residual plastic deformation of TPU and irreversible damage of the electrically conductive MWCNT network. In the second and following cycle series the difference between responses is not such significant comparing with the first cycle series. Similar strain stabilization effect is observed for preloaded specimen when in consecutive cycles the relative resistance change or irreversible part are nearly equal

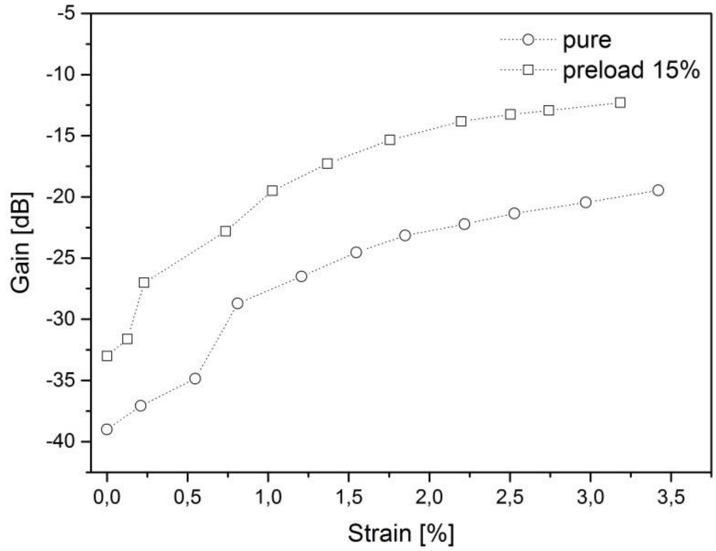

Fig. 3 Dependence of the gain change determined for MWCNT/PU laminate on applied strain. Pure MWCNT/PU laminate (cycle). Applied strain in the range from 0 to 3.5 %. 15 % Preload sample (square). Applied strain in the range from 0 to 3.25 %.

After specimen loading, deformation of polyurethane/CNT laminate specimen increase. This behavior is monitored by gain. The gain is decreasing when the specimen is loading. The gain decreasing is caused by changing resistivity of measured specimen. The carbon nanotubes network sensitively reacts to applying strain Fig. 3.

For simulation of damage construction 15% preload was applied. Then the similar strain in the range fom 0 to 3.25% was applied. Preloading caused decrease the gain of tested specimen. The shape of the curve strain vs. gain of preloaded sample is similar. This fact clearly shows destruction of monitored construction. Decreasing of gain is caused of crack formation in the CNT network after preloading. The crack formation increase the sensitivity of CNT network due to increasing the contacts point in the network Fig. 3.

Conclusions

High elastic polyurethane/carbon nanotubes laminate can be used as a sensing element for deformation detection. Sensing element works as an antenna with stable resonant frequency 650 MHz. For the testing sensing element was applied extension/relaxation cycles. The cycle deformation gradually increases in each 5 minutes cycle up to maximal strain value approximately 3.5 %. Ten consecutive extension/relaxation cycles was applied. During the applying each cycle also the gain response was measured. The same sample was 15% preload to simulate destruction of monitored construction. . Than the strain in the range from 0 – 3.25% was applied. The change of gain is visibly different. This fact clearly shows damage of monitor construction. The principle of function can be described as a change of resistivity of carbon nanotubes network during applying strain. The presented sensor can be used for structure health monitoring(SHM). This type of nondestructive inspection of monitored construction is very important of permanently used construction for instance bridges, towers or blades of wind power plant. This sensing element is made from polymer, very sensitive to applied strain, small in size.

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References

[1] I. Kang, Y.Y. Heung, J.H. Kim, J. Won Lee, R. Gollapudi, S. Subramaniam, et al. Composites: Part B, 37 (2006), pp. 382–394
[2] Kang I., Schulz M.J., Kim J.H., Shanov V., Shi D. A carbon nanotube strain sensor for Structural health monitoring. Smart Mater. Struct. 2006;15:737–748.
[3] Yamada T., Hayamizu Y., Yamamoto Y., Yomogida Y., Izadi-Najafabadi A., Futaba D.N., Hata K. A stretchable carbon nanotube strain sensor for human-motion detection. Nat. Nanotechnol. 2011;6:296–301.
[4] C.L. Li, E.T. Thostenson, T.W. Chou Compos Sci Technol, 68 (2008), pp. 1227–1249