IOP Conf. Series: Materials Science and Engineering **731** (2020) 012001 doi:10.1088/1757-899X/731/1/012001

Investigation of the Structure and Properties of SWCNT-Modified Epoxy Specimens

Burkov M V[1,2] and Eremin A V[1]

[1]Institute of strength physics and materials science, Akademicheskiy avenue 2/4, Tomsk, 634055, Russia
[2]Tomsk Polytechnic University, Lenin avenue 30, Tomsk, 634050, Russia

burkovispms@mail.ru

Abstract. SWCNT-modified epoxy with advanced electrical properties was investigated in the paper. CFRPs have high specific strength and elastic modulus which are the most important properties for aerospace engineering. However a lack of electrical and heat conductivity makes efficient lightning strike protection and thermal de-icing complicated. In this research electric conductivity of epoxy binder for CFRP production is modified by addition of single-wall carbon nanotubes. Measurements of electrical conductivity of hardened epoxy binders are performed showing good increase of conductive properties.

1. Introduction

Nowadays the application of composites has increased drastically. Being the materials composed of two or more constituents they provide an unlimited variety of physical and mechanical properties that fit the exact application demands. One of the modern trends in the field of engineering of composites for structural applications is related to combined reinforcing by fiber-based materials (fabrics, mats, etc) and different additives (microfibers, nanofillers, etc). This allows obtaining better mechanical properties and achieving new quality that is not inherent to traditional fiber-reinforced polymers (FRP). For example one approach for providing electrical conductivity to carbon FRP is addition of carbon nanotubes (CNT) resulting in so-called hybrid CFRP [1,2].

There are different additives being studied to modify FRPs. Metal powders can be used for providing electrical conductivity and improving thermal conductive properties [1,3,4]. Ferromagnetic particles are applied for the production of electromagnetic shielding composites [1,5]. Different research groups use microsized additives and nanomaterials [6,7] concluding that both types can be used for manufacturing of hybrid FRPs having advantages and disadvantages.

One of the technological problems which should be solved when designing a hybrid CFRP is the homogeneous distribution of small-sized reinforcement in the matrix (thermoset or thermoplastic). In the case of microfillers like short milled fibers of metal powders this problem is often solved by using such techniques like manual stirring or mechanical mixing. But as for nanomaterials the dispersing of particles is quite difficult resulting in the necessity of application of high-energy mixing procedures [8,9]. One of the robust techniques is ultrasound (US) assisted mixing, which can be used both in laboratory conditions and in industrial environment. There are a lot of scientists investigating US treatment for production of different advanced materials.

The technology of mixture preparation is often limited only to laboratory conditions and equipment resulting in high costs and low productivity. Thus it is quite important to search for novel methods that will be both efficient and cost-effective. The paper is devoted to the investigation of structural features and mechanical properties of SWCNT-modified epoxy specimens obtained under different conditions.

2. Materials and methods

The specimens were prepared in order to investigate structural properties, compressive strengthand electrical conductivity. Single-wall carbon nanotubes used are provided by OcSiAl (Russia, Novosibirsk) having an outer mean diameter of 1.6±0.4 nm and a length of more than 5 µm. The residual impurities and moisture are less than 15 and 5 wt.% correspondingly. Epoxy resin of L grade and hardener CL were supplied by R&G, Germany. This epoxy binder (L epoxy + CL hardener) can be used to produce medium-to-high performance CFRPs which have the properties comparable to CFRP made using aerospace-grade epoxies.

The mixtures of epoxy and SWCNT were prepared by mechanical mixing during 1 minute and ultrasonication using UZDN-A (Russia) probe sonicator for 20 minutes with a frequency of 22 kHz and power of 130 W. During ultrasonic mixing the glass was constantly cooled in water. After cooling the hardener was added to the mixture and poured into molds for preparation of cylindrical specimens with a diameter of 12.7 mm and a length of 30 mm. Summary there were initial non-modified epoxy specimens and 3 types of SWCNT-modified with 0.1 wt.%, 0.2 wt.%, and 0.3 wt.% of SWCNT. In order to perform conductivity measurement the specimens were coated with silver conductive paint. The paint was applied on the ends of the cylinder and electrical conductivity was measured between these pads using LCR meter E7-20, Russia.

After measurement of electrical properties the specimens were tested by uniaxial compression using Instron 5582 universal testing machine at the rate of 2.5 mm/min. Three specimens of each CNT content were prepared. The fracture surfaces of the specimens were investigated by SEM using LEO EVO 50 (Zeiss, Germany) in "Nanotech" ISPMS SB RAS.

3. Experimental results and discussion

3.1. Electrical conductivity

The results of conductivity measurement are presented in Figure 1 and Table 1. Non-modified epoxy has nearly zero electrical conductivity: the upper threshold of resistance measurement of the used device is 10^9 Ohm while the measured resistance exceeds this threshold resulting in electrical conductivity less than 10^{-7} S/m.

The addition of 0.1% of SWCNTs increases the conductivity to 0.012 S/m. Larger content of SWCNTs results in nonlinear (Figure 1) rise of conductivity to 0.063 S/m for specimen CNT-3. The scatter of the conductivity for CNT-1 is about 6% while for CNT-2 – 27%. The obtained results demonstrate the possibility of enhancing the electrical conductivity of different materials using SWCNTs: the dielectric epoxy after modification acquired conductive properties.

Table 1. Electrical conductivity of specimens.

Specimen	Electrical conductivity, S/m
CNT-0	$<10^{-7}$
CNT-1	0.012±0.0007
CNT-2	0.03±0.0083
CNT-3	0.063±0.0025

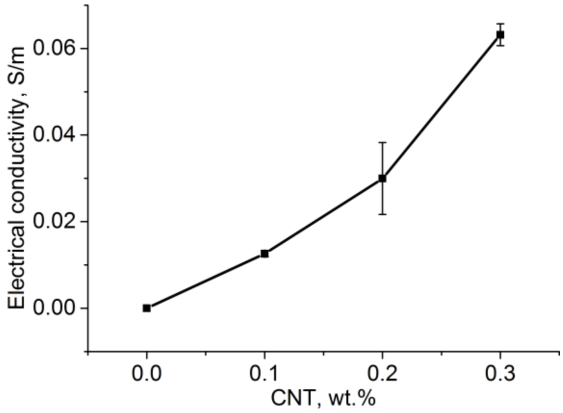

Figure 1. Results of conductivity measurements of epoxy specimens.

3.2. Compression testing

Because the viscosity of epoxy due to addition of SWCNTs drastically increases thus final porosity of epoxy specimens with SWCNTs has risen. The unmodified epoxy has low porosity and compression strength of ~111 MPa. The addition of 0.1% and 0.2% of SWCNTs results in decrease of compression strength to 87 and 63 MPa consequently. For specimen CNT-3 it was decided to use degassing procedure which resulted in decrease of porosity leading to better compressive strength – 105 MPa.

3.3. Scanning electron microscopy

Figures 2, 3 and 4 show SEM images of fractured epoxy specimens CNT-1, CNT-2, and CNT-3 respectively. It can be seen from these results that the specimen CNT-1 fractures with mainly viscous behavior. The porosity of the specimen is low, but compared to the non-modified epoxy there is a drop in compressive strength (87 MPa versus 111 MPa) which leads to the emergence of pores.

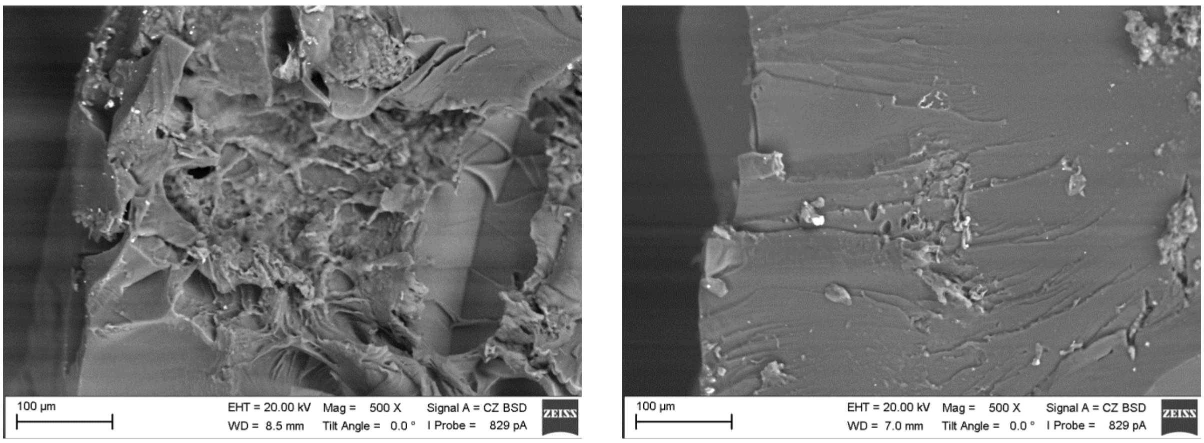

Figure 2. SEM images of fractured CNT-1 specimen.

Larger content of SWCNTs of the CNT-2 specimen results in the remarkable increase in viscosity making it difficult to homogeneously mix the epoxy with hardener without the introduction of air in the mixture. Small air bubbles combine in large pores during molding and there are easily seen in the fracture surfaces of the CNT-2 specimen (Figure 3). The fracture behavior transits to the more brittle one and along with porosity the mean compressive strength drops to 63 MPa.

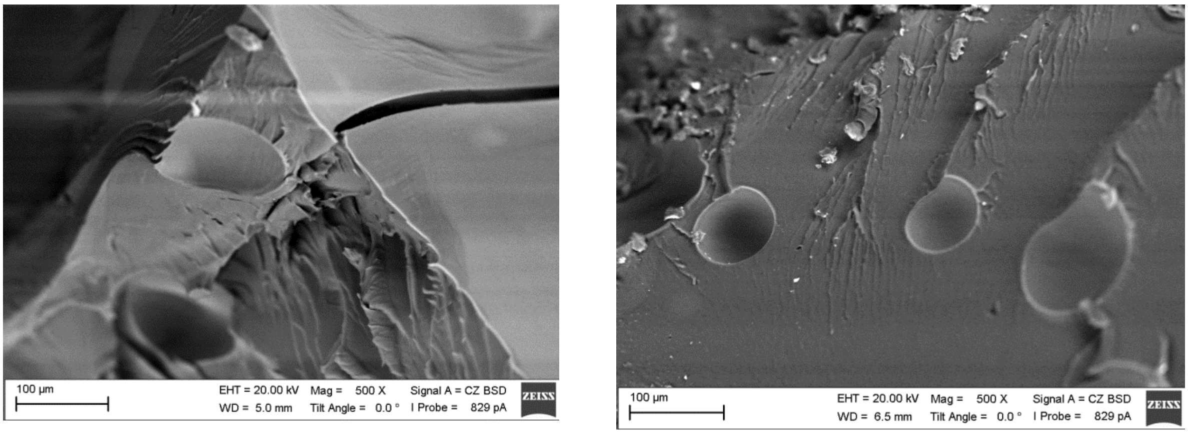

Figure 3. SEM images of fractured CNT-2 specimen.

As can be seen from the SEM images of the CNT-3 specimen the structure contains fewer pores than the structure of CNT-1. It is due to the use of a degassing procedure which resulted in decrease of porosity and preservation of compressive strength at a level of neat epoxy (105 MPa). The fracture behavior is even more brittle.

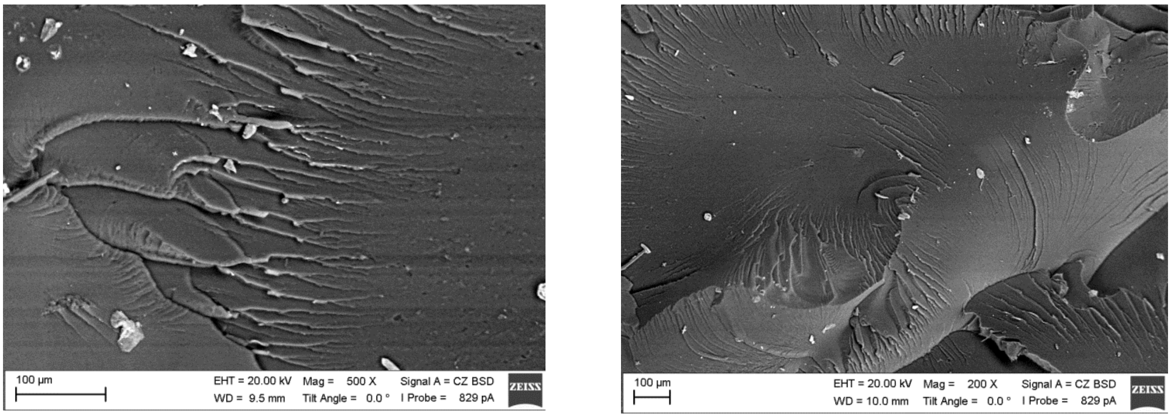

Figure 4. SEM images of fractured CNT-3 specimen.

4. Conclusion

The SWCNT modified epoxy was investigated. The research was focused on the evaluation of electrical conductivity and compressive properties. Electrical conductivity after the addition of SWCNT rose from null values to 0.063 S/m. It is found that the viscosity during addition of SWCNT increases drastically thus the degassing procedure is obligatory. Otherwise significant porosity is emerged. The method proposed in the work for producing hybrid composites has good potential and future research is to be linked to the development of reliable and inexpensive preparation and mixing techniques for carbon fiber reinforced polymers.

Acknowledgements

The research was supported by Grant of Russian Science Foundation №19-79-10148.

References

[1] Gibson R F 2010 A review of recent research on mechanics of multifunctional composite materials and structures *Compos. Struct.* **92** 2793–810

[2] Tehrani M, Safdari M, Boroujeni A Y, Razavi Z, Case S W, Dahmen K, Garmestani H and Al-Haik M S 2013 Hybrid carbon fiber/carbon nanotube composites for structural damping applications *Nanotechnology* **24** 155704
[3] Cortes L Q, Racagel S, Lonjon A, Dantras E and Lacabanne C 2016 Electrically conductive carbon fiber / PEKK / silver nanowires multifunctional composites *Compos. Sci. Technol.* **137** 159–66
[4] Jang J, Park H C, Lee H S, Khil M-S and Kim S Y 2018 Electrically and Thermally Conductive Carbon Fibre Fabric Reinforced Polymer Composites Based on Nanocarbons and an In-situ Polymerizable Cyclic Oligoester *Sci. Rep.* **8** 7659
[5] Zhao Z J, Zhang B Y, Du Y, Hei Y W, Yi X S, Shi F H and Xian G J 2017 MWCNT modified structure-conductive composite and its electromagnetic shielding behavior *Compos. Part B Eng.* **130** 21–7
[6] Panin S V., Duc Anh N, Kornienko L A, Alexenko V O, Buslovich D G and Ovechkin B B 2018 Wear-resistant polyetheretherketone composites *Mater. Today Proc.* **5** 25976–82
[7] Panin S V., Le T M H, Kornienko L A, Alexenko V O, Ivanova L R and Ovechkin B B 2018 Mechanical and tribotechnical properties of polyphenylene sulfide composites reinforced with carbon fibers of various dimension p 020231
[8] Caneba G T, Dutta C, Agrawal V and Rao M 2010 Novel Ultrasonic Dispersion of Carbon Nanotubes *J. Miner. Mater. Charact. Eng.* **09** 165–81
[9] Kim M and Kim J 2018 Nanoparticle dispersionizer by ultrasonic cavitation and streaming *Jpn. J. Appl. Phys.* **57** 07LE03