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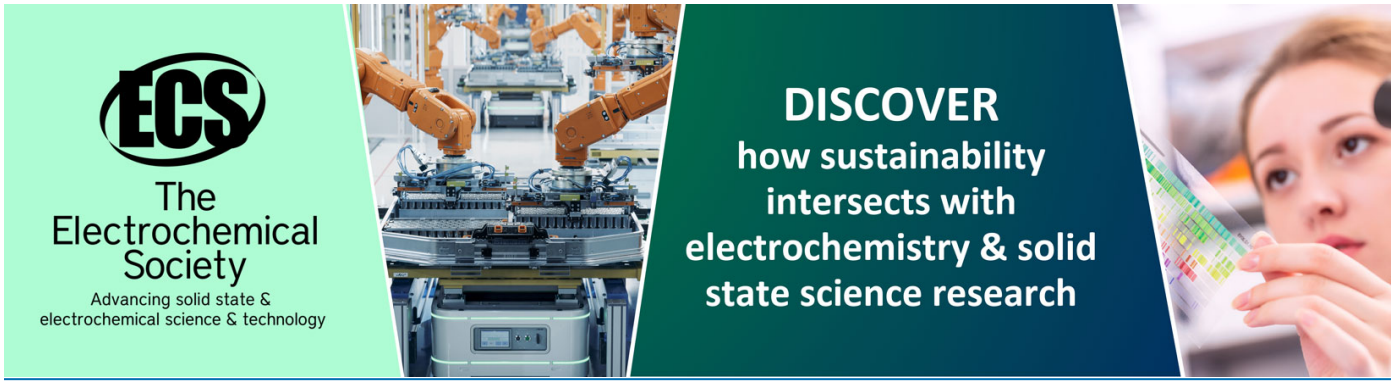

Studies on structural MWCNT/epoxy nanocomposites for EMI shielding applications

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Abstract. This paper presents the preparation and characterization of MWCNT - Multi-Walled Carbon Nano-Tube/epoxy nanocomposites, as materials with a good yield for shielding electromagnetic interferences - EMI. Both the precursors used, and the nanocomposites obtained were characterized by different techniques: FTIR - Fourier transform infrared spectroscopy, Raman spectroscopy, SEM - Scanning Electron Microscope and XRD - X-ray diffraction. The electrical and mechanical properties of the nanocomposites were estimated compared to the epoxy resin without addition. Electromagnetic interference shielding tests were conducted in the 2.5 - 6.4 GHz frequency band. The results were found to be promising since for the MWCNT/epoxy sample it was obtained a 20 dB attenuation when compared with the ambient air.

1. Introduction

The replacement of carbon fibers in the structure of polymeric nanocomposites with carbon nanotubes was an important step in the field of nanotechnology [1]. The emergence of carbon nanotubes [CNT] has opened up important areas of applicability in the electronics industry, due to the thermal and strengthening properties at the nanometer scale [2], the electronic properties [3] and the superior chemical resistance [4, 5]. Due to the structure of single-cylinder or multi-cylinder graphene with a high aspect ratio and low density, MWCNT has special mechanical and electrical properties [6]. The special properties of CNT such as adhesion, chemical resistance and dimensionless stability contribute to obtaining epoxy nanocomposites with applicability in the automotive, aerospace, adhesive encapsulation, anticorrosive coatings, or the manufacture of materials with electromagnetic shielding properties. The hardening kinetics and chemical composition of epoxy resins influence the properties of nano-epoxy materials such as: Young's modulus, density, electrical and thermal conductivity. In the process of preparing CNT-epoxy nanocomposites, the liquid medium consisting of an epoxy or amine component, incorporates carbon nanotubes, leading to a good dispersion of them, then proceeds to the

ultrasonic homogenization procedure [7], hardening of the resulting dispersion, by adding the resulting hardener composite in solid state. The amine hardener facilitates the opening of the epoxy resins ring and due to the reaction of the two components, the molecular opening of the epoxy resins ring and due to the reaction of the two components, the molecular mass increases, which implicitly leads to the hardening of the composite. In order to obtain certain properties of the CNT/epoxy nanocomposite, we use the functionalization processes [8] of nanotubes that control the rheological properties of the liquid composition or the Van der Waals bonds between the nanotubes and the liquid component [9]. Methods used in recent applications include: matrix use of carbon nanotubes with other nanoparticles [10, 11], nanotube surface functionalization [12], use of wetting surfactants [13], efficient methods for mixing and dispersing nanotubes in the liquid epoxy component [14], the use of mixed epoxy/copolymer nanocomposites [15] or fillers in combined form using nanotubes/graphene [16 - 20]. The use of a resin/hardener with low viscosity represents a major advantage in the process of preparing epoxy nanocomposites, which leads to the incorporation of a large amount of CNT, without modifying their rheological properties. The increase of the viscosity in case of a large specific surface leads to the modification of the structural properties of the nanocomposite, due to the inhomogenization of the dispersion and the incorporation of air particles. The following is a working method for obtaining the MWCNT dispersion in the epoxy system.

In this paper the electromagnetic waves shielding potential of some composite materials is evaluated. The analyzed composites show the efficiency of the electromagnetic waves in the 2.5 – 6.4 GHz band, which proves the utility of these materials in making some of the electromagnetic screens in environments in which the use of materials for classical screens (metals) is forbidden.

2. Materials and method

The epoxy component Epo-tek® 301 (Epoxy Technologies, Inc) is used as the polymer matrix, in liquid form, consisting of two components: the epoxy component (A) and the amine component (B). Carbon nanotubes (MWCNT –BPD30L520, NanoLab) having a length between 5-29 μm, purity > 95%, and an average diameter of 30 nm.

2.1. Preparation method of MWCNT / epoxy nanocomposites

In order to characterize the materials (MWCNT/epoxy nanocomposites and epoxy polymer), specimens were made in the form of discs. The discs were made by casting in PVC (PolyVinyl Chloride) molds arranged on PET (Poly-Ethylene Terephthalate) lamination foils, supported by glass supports (figure 1), and for the stripping of the materials was used silicone Vaseline (High vacuum silicone grease – Dow Corning). Carbon nanotubes (MWCNT) are weighed, mixed, and homogenized in epoxy component (A), which with low viscosity (100-200 cP) makes it possible to easily incorporate materials with a large specific surface area.

Figure 1. Precursors and samples of MWCNT/epoxy nanocomposites. From left to right: epoxy resin (A) (Epotek E301), amine hardener (B) (Epotek E301) and cured samples on nanocomposites.

A MWCNT/epoxy mixture is obtained and is subjected to the ultrasonic procedure for one hour in the bath (Elmasonic 300), using frequencies in the range of 25/45 kHz. The MWCNT/epoxy component thus obtained is mixed with the amine component (B) in a gravimetric mixing ratio of 4:1. The mixture is made at room temperature for 15 minutes, then 30 minutes for pre-reaction, stationary for 30 minutes. The resulting composition is poured into PVC discs in which it hardens for 24 hours. A gelation period of 2 hours was used, with a relative humidity of 50% and a temperature of 220ºC. Then for 5 minutes the specimens are ultrasound in isopropyl alcohol (VLSi degrees, BASF), to remove the silicone grease and then follow the drying time. After stripping from the molds, 7 days of maturation follow, then the tests are performed on the respective specimens.

It was opted for concentrations of MWCNT of 0.3% and 0.6%, because MWCNT present large specific surface, which leads to the increase of the formed nano-composite viscosity. At high MWCNT concentrations is very difficult to homogenize the epoxy component (A) as well as hard to incorporate the amine component (B) for hardening and difficult to homogenize the formed (A + B) mixture. It was observed that at MWCNT concentrations lower than 1% in the nanocomposite, the percolation phenomenon exists and the unhardened mixture (A + B) can be easily poured in the molds without the occurrence of structural defects.

2.2. Methods for characterization

All used materials, the starting substances, and the obtained nanocomposites, were characterized by different methods: X-ray diffraction, scanning electron microscopy (Nova NanoSEM 630), Raman spectroscopy, infrared spectroscopy. Mechanical tests, electrical resistance measurements and electromagnetic shielding tests were also performed.

Diffraction spectra were studied using the diffractometer (Rigaku Smartlab 9kW rotating anode).

IR analysis was performed on FTIR equipment (BRUCKER27). The FTIR Tensor 27 spectrometer (Bruker Optics), and the ATR (attenuated total reflectance) with Platinum accessory allowed the study of the structural transformation of the raw materials up to the composite stage. A scan speed of 64 scans was used and spectra were drawn over a range between 4000 and 400 cm^{-1}.

Scanning electron microscopy was used to morphologically characterize the obtained carbon nanotubes and nanocomposites. For analysis, a small amount of carbon nanotubes arranged on a conductive strip is used. The disks were cooled in liquid nitrogen and then fractured for morphological analysis of MWCNT/epoxy nanocomposites and highlighting their surface. Due to the dielectric properties of the epoxy material on the resulting small pieces a gold layer of 4 − 5 nm was deposited.

For Raman spectra a high-resolution equipment (LabRAM HR800 Horiba) was used.

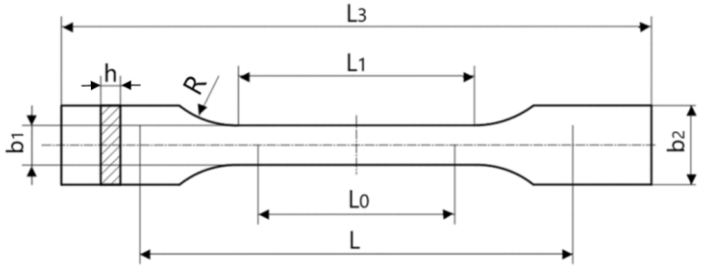


Figure 2. Dimensions of the tensile test specimen.

In figure 2 there are presented the dimensions of the tensile test specimen (type 1B) according to SR EN ISO 527-4: 2000 [21]: the total length (L_3 = 160 mm), length of narrow parallel part (L_1 = 60mm), radius (R = 60mm), width of extremities (b_2 = 20mm), width of the narrow part (b_1 = 10mm), thickness (h = 2.5mm), gauge length (L_0 = 50mm), initial distance between grips (L_3 = 114mm).The test system with which the mechanical measurements were performed, Mecmesin MultiTest 2,5i, with high performance load cells (ILC) of 2.5 kN, makes the obtained results to have very good accuracy

and resolution. The precision mechanical system has a positioning error of ± 130 μm at a displacement of 300 mm. The Emperor software package provides a large programming capacity with many analysis options which leads to a very good characterization of the samples under analysis. The mechanical tests were performed on 3 material samples: MWCNT 0.3% / epoxy, MWCNT 0.6% / epoxy and epoxy resin (epoxy). The standards used are in accordance with the tested materials and to obtain comparative results in the three materials.

During the tensile tests performed, an axial force was applied to the tested specimen, until the material failed. The maximum traction force was 500 N and the gripping speed of the grips was 5 mm/minute. The test conditions for the 3 samples were identical. Calibration operations were performed on the measuring cell at the beginning of each test.

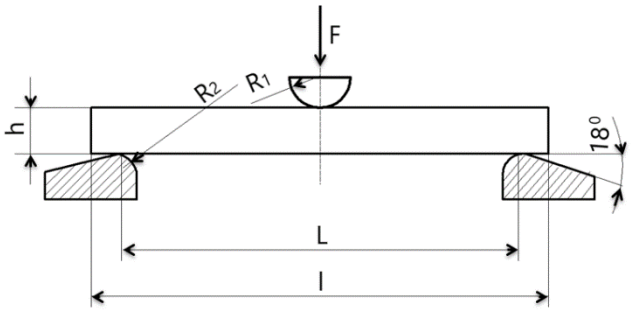


Figure 3. Dimensions of the flexure test specimen.

In figure 3 there are presented the dimensions of the flexure test specimen according to SR EN ISO 178:2019 [22]: length (l = 50 mm), support span (L = 40mm), width (b = 25 mm), thickness (h = 2.5 mm), radius of loading edge (R_1 = 5 mm), radius of supports (R_2 = 2 mm).

The specimens subjected to the flexure tests were subjected to forces perpendicular to the axis and to the half of the length of the specimen, registering the variations appeared, until the failing of the material.

The electrical resistance tests were performed using the Hioki 3532-50 LCR HITESTER device, with a frequency range of 42 Hz to 5 MHz, a response time of 5 ms, and ± 0.08% accuracy. The test samples had a length of 25 mm and a section of 12×5 mm^2. For each sample, tests were performed for three measurement frequencies.

EMI shielding of an electronic device is achieved by attenuating the passage of electromagnetic waves by reflection or by the absorption of the power of the incident ray. Due to the tendencies towards miniaturization of electronic devices, lightweight materials have been developed to replace metals with improved properties including corrosion resistance, flexibility, and speed in processing [18].

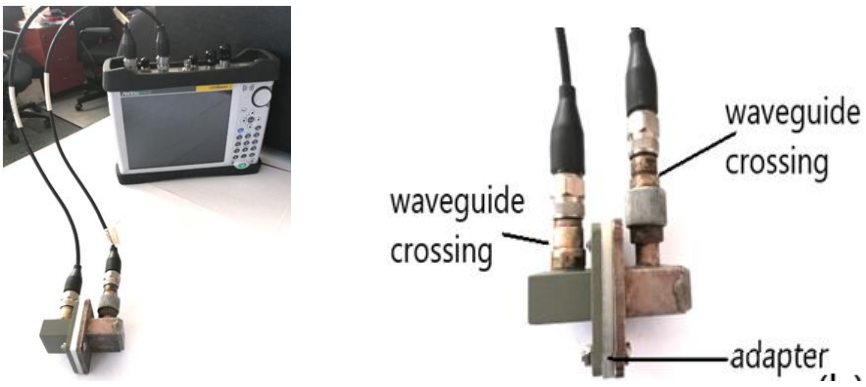


Figure 4. (a) Anritsu S 412 wave analyzer;
(b) Detail of waveguide switch adapter assembly.

A high-performance Anritsu S 412 vector network analyzer (figure 4a) with a 500 kHz to 6,400 MHz bandwidth was used in order to verify the attenuation introduced by the analyzed samples. Each sample was mounted in the adapter attached between the two coaxial cable-guide couplings type PVK 35X15, with a cut-off frequency of 2.5 GHz (figure 4b).

Tests were performed on three material samples, neat epoxy sample, 0.3% MWCNT/epoxy nanocomposite and 0.6% MWCNT/epoxy nanocomposite respectively, having the following dimensions: length = 35 mm, width = 15 mm, thickness = 3.5 mm. In the preparation process of samples upon mixing a sonication steps [19], debundling of MWCNT is effective without altering their length, leading to better EMI shielding efficiency [20].

3. Results and discussions

The absorption bands that are assigned to the C−H groups (figure 5) from the precursor components (aliphatic and aromatic), characterize the spectrum of the epoxy component (A).

The spectral bands 2970-2850 cm^{-1} confirm the existence of methyl and methylene groups and the peak centered at 3055 cm^{-1} confirms the C−H bonds in the epoxy and benzene ring. The C-O bonds in the range 1230 − 830 cm^{-1} belong to epoxy precursor (A). The C−O−C epoxy group in precursor (A) is assigned the peak centered at 913 cm^{-1} and is considered the imprint of this group. At approximately 3447 cm^{-1} there is a low intensity band that shows the existence of O−H bonds which confirms the presence of dimers or compounds with high molecular weight or phenolic groups. The bands at 1607 and 1582 cm^{-1}, which belong to the C−C bonds in the benzene skeleton, confirm the benzene ring in the phenolic component. The peaks of the N−H bonds (3367, 3290 and 817 cm^{-1}) show the presence of the amine component (B). At 1013 cm^{-1} is found the absorption peak that defines the C−N group. At 1460 and 1378 cm^{-1} are found the peaks of the methyl, methylene (−CH3, −CH2) groups. In the spectrum of the sample there are also peaks that represent the vibration mode of the C−H, C−N and C−O bonds. In the spectrum that characterizes carbon nanotubes, there are no bands that show their functionalization. FTIR spectra for final products: hardened MWCNT/epoxy and epoxy (A + B) nanocomposites come to prove the respective reactions by the disappearance of the bands that would show the presence of C−O−C from epoxy (from 913 cm^{-1}) attributed to component (A) and N−H bonds attributed to component (B). In the area 4000-3000 cm^{-1} there is a wide band that shows the formation of O−H bonds in the polymer and the crosslinked composite.

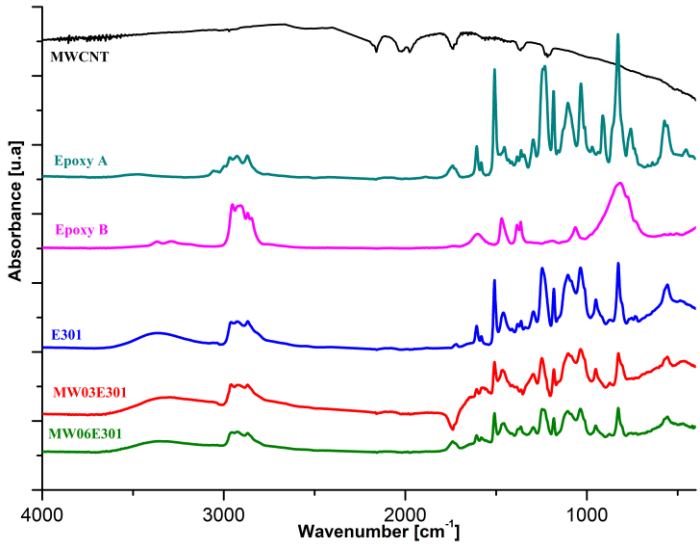
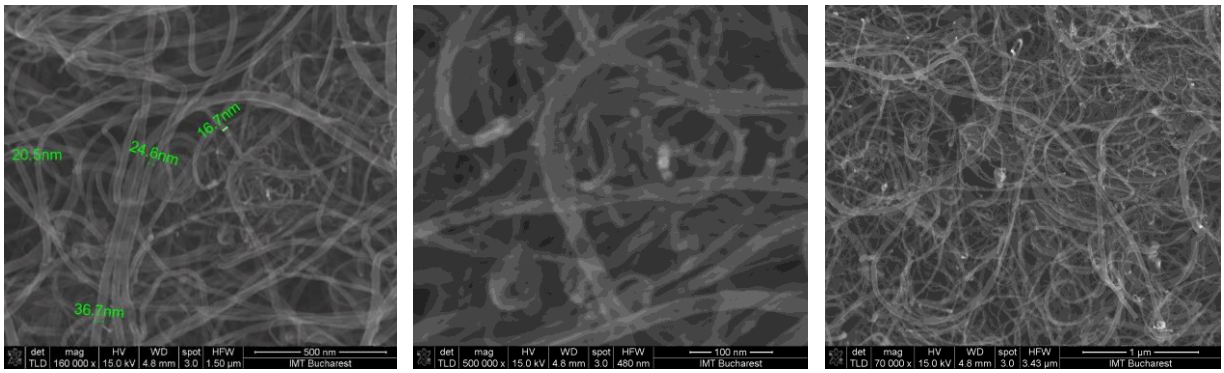


Figure 5. FTIR spectra for precursors consisting of solid cured epoxy and amine and MWCNT; hardened components made of epoxy (E301) and nanocomposites at concentrations of 0.3% (MW03E301) and 0.6% (MW06E301) respectively.

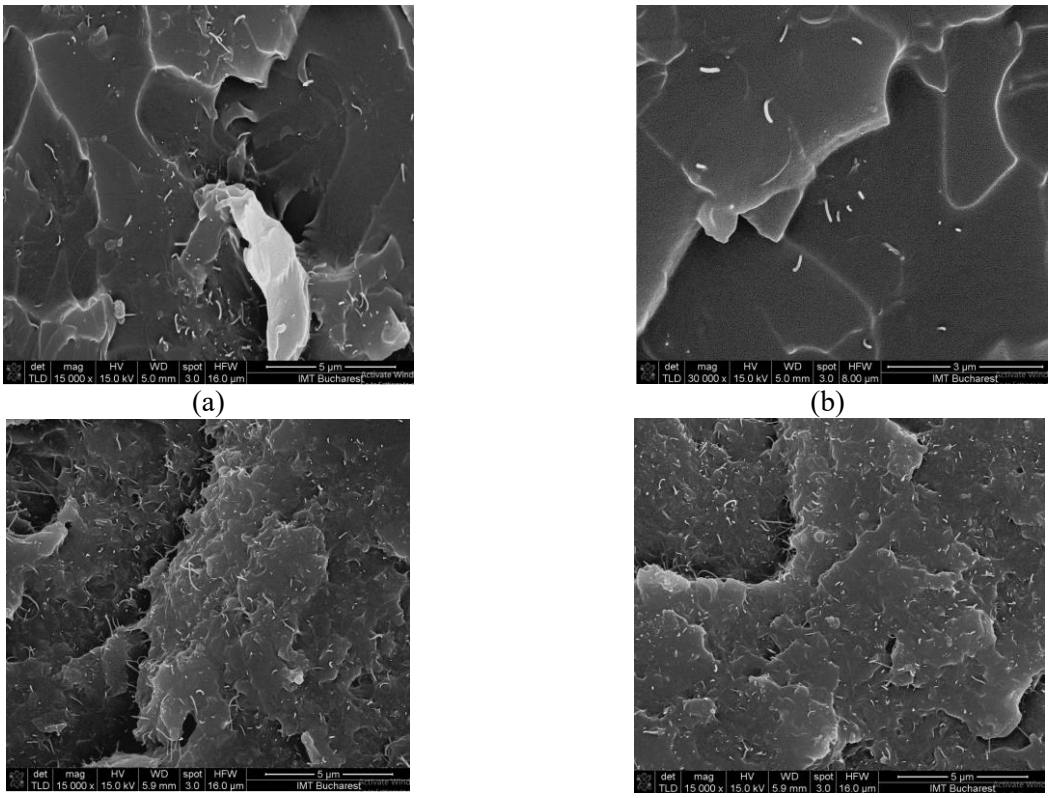
Magnifications of 70000×, 160000× and 500000×, respectively, of carbon nanotubes are shown in figure 6. A coiled filiform structure is observed, the fibers are randomly oriented and have a high aspct ratio.Carbon nanotubes have a diameter of 10-35 μm.



(a) (b) (c)

Figure 6. MWCNT at resolutions of 70000 x (c), 500000 x (b) and 160000x (a) respectively.

Figures 7a and figure 7b show the image of SEM MWCNT/epoxy nanocomposites, filler concentration of 0.3% for a magnification of 15000x, respectively 30000x. Figures 7c and figure 7d show the image of SEM MWCNT/epoxy nanocomposites, filler concentration of 0.6% for a magnification of 15000x. The fractured epoxy matrix shows a homogeneous distribution and a dense morphology. The appearance is slightly lamellar, and the carbon nanotubes show a uniform dispersion in the polymer matrix.



(a) (b)

(c) (d)

Figure 7. MWCNT/epoxy nanocomposites: (a, b) filler concentration of 0.3%;
(c, d) filler concentration of 0.6%.

The results of Raman spectrometry for carbon nanotubes (MWCNT) are shown in figure 8. The Raman spectrum in which the bands G and D located at 1582 cm^{-1} (band G) confirm a graphical structure, the peak at 1327 cm^{-1} (band D) shows the vibration of the C−C bonds that are activated in case of defects in the graphical system. There is a wide band having order 2 (G'), having low intensity, proportional to the increase of the density of the defects being at 2652 cm^{-1}.

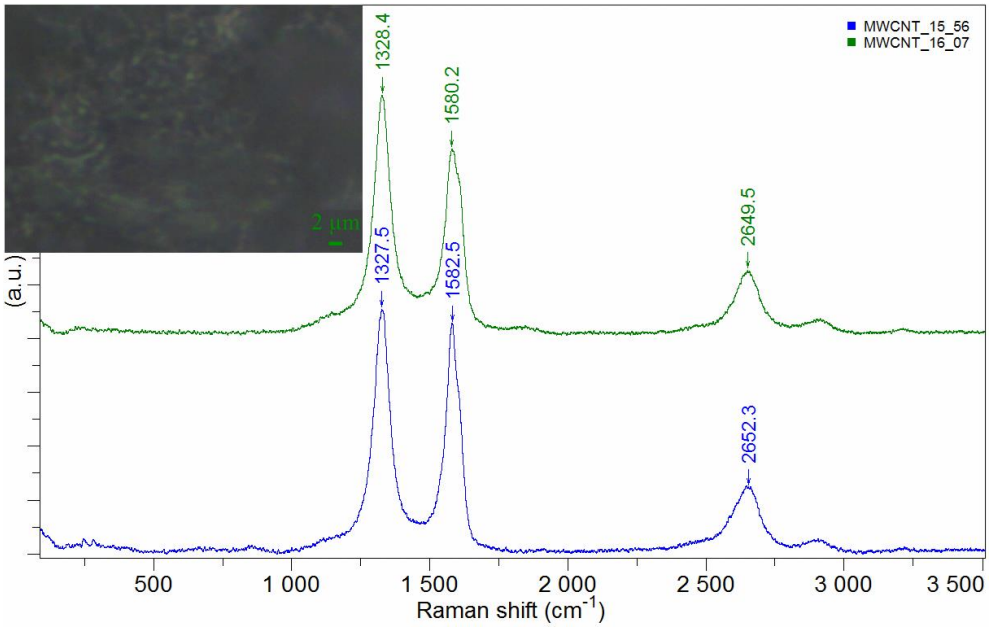


Figure 8. Raman spectrum for MWCNT before sonication. I nset picture: surface of analyzed sample.

The diffraction spectra resulting (figure 9) from the X-ray analysis, for the MWCNT/epoxy and MWCNT nanocomposites, show a wide diffraction peak due to orientations (101) and (100) in the range 43 − 450 and a peak that is at 26° having FWHM (Full width at half maximum) = 1.6°. Following the dispersion in the epoxy network, the MWCNT/epoxy nanocomposites, have two wide bands at 20° and 43°, which shows the existence of an amorphous structure. In the case of low concentration composites, the characteristic peak for MWCNT is not recorded in the analysis framework.

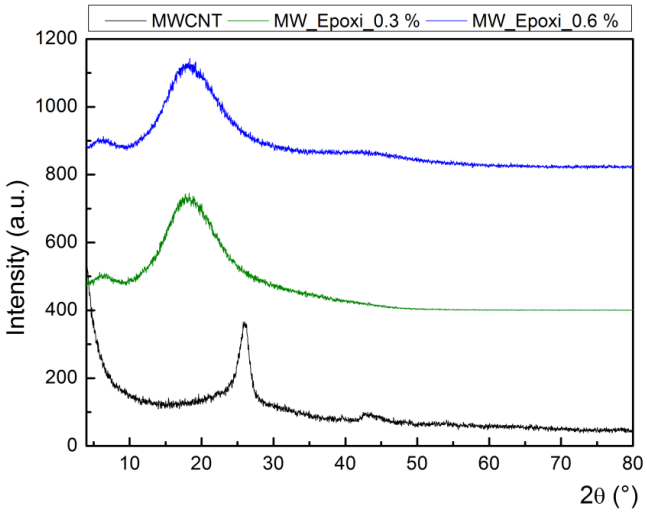


Figure 9. XRD spectra for MWCNT and epoxy / MWCNT nanocomposites.

The test piece made of epoxy resin, according to figure 10, went through the area of elastic deformation, following an approximately linear evolution, up to a force of 350 N. The material underwent an elongation of 3 mm, then entered the falling area (plastic deformation) and failed at an elongation of 3.3mm. In the case of the 0.3%/epoxy MWCNT sample, the elastic zone is increased to a force of 400 N, the material elongating by 2.8 mm, then the plastic deformation follows up to an elongation of 4.2 mm, following the rupture of the specimen. With the addition of 0.3% MWCNT can be seen an increase in tensile strength, the area of elastic and plastic deformation increases. For the MWCNT 0.6%/epoxy sample, an increase of the elastic deformation area can be observed. At an elongation of 7 mm the material fails. In conclusion, which makes it usable as an insulator, in elasticity and ductility of the respective material, which makes it usable as an insulator, in installations, electrical cables, in high voltage work areas. Tensile tests were performed according to SR EN ISO 527-4: 2000 [21].

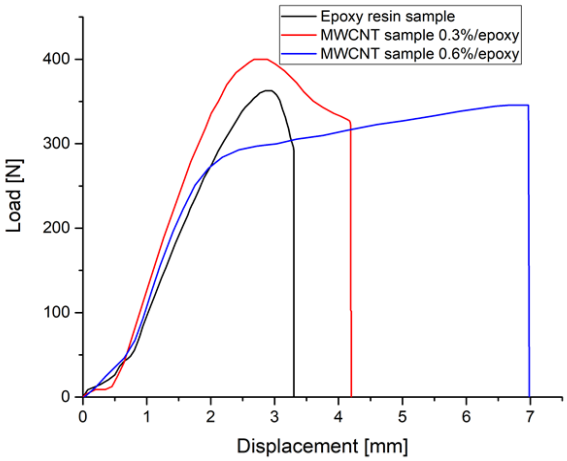


Figure 10. Tensile strength diagram for neat epoxy, 0.3% MWCNT composite and 0.6% MWCNT composite sample, respectively

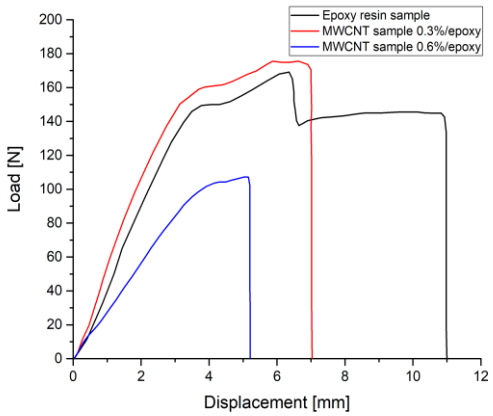


Figure 11. Flexure stress resistance diagram for epoxy resin sample, 0.3% MWCNT composite and 0.6% MWCNT composite, respectively.

From the obtained results according to figure 11, in the flexure of the specimens, it is found that the presence of MWCNT has an influence on the binding forces between the molecules of the respective polymer. As the amount of MWCNT increases, the intermolecular bonding forces decrease, the plastic deformation zone decreases. Flexure tests were performed according to SR EN ISO 178:2019 [22].

The results are presented in table 1 where it is observed that the increase of the MWCNT composition leads to an exponential decrease of the electrical resistivity.

Table 1. Measured values of electrical resistance for the three measurement samples.

Frequency	Neat epoxy	0.3% MWCNT /epoxy	0.6% MWCNT /epoxy
f = 50 Hz	>1 GΩ	>1 GΩ	0.35 MΩ
f = 800 Hz	0.9 GΩ	40 MΩ	3 MΩ
f = 8 kHz	373 MΩ	10 MΩ	0.9 MΩ

The attenuation produced by the analyzed samples increases proportionally with the concentration of MWCNT present in the epoxy matrix. According to the results of figure 12 it was found that in the 2.5 GHz and 6.4 GHz band, the attenuation introduced by sample around the 5.5 GHz central frequency is approximately 20 dB between ambient air and 0.6% MWCNT/epoxy sample.

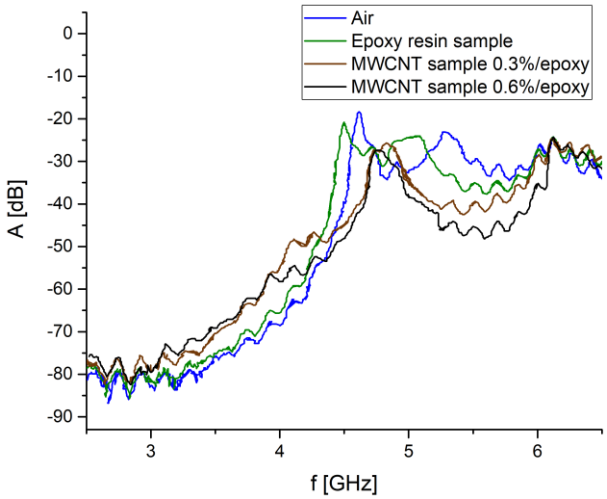


Figure 12. Attenuation produced by samples of neat epoxy, 0.3% MWCNT/epoxy and 0.6% MWCNT/epoxy, respectively.

4. Conclusions

Epoxy nanocomposites with carbon nanotubes were prepared using specific and easy methods. Two concentrations of 0.3% and 0.6% MWCNT embedded in epoxy resin were used. The characterization of the newly obtained materials reveals in content a homogeneous and flawless morphology. Mechanical tests show an increase in the tensile strength of newly formed nanocomposites compared to the epoxy material. In the case of the electrical resistance of nanocomposites, this decreases with the increase of the amount of MWCNT. In the case of EMI shielding tests, the attenuation was proportional to the amount of MWCNT present in the nanocomposite with a maximum value of 20 dB.

Acknowledgements

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