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The Role of Reduced Graphene Oxides in Enhancing the Mechanical Properties of Satellite Structure Nanocomposite Materials against Electron Beam

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Abstract

Materials developed for space application to sustain both of mechanical loads ant space environmental threats. The Electron Beam (EB) was selected to represent one of the most hazardous space environment parameter subjected to the Spacecraft (SC) components; the charged particles flux which predicted according to the space mission and its orbit parameters. The candidate materials for this study based on the carbon fiber/epoxy which was enhanced by adding Reduced Graphene Oxide (RGO) and investigate its resistance to EB. RGO addition was varies in three different compounds with different preparation methods; (RGO-24N, RGO-33C and RGO-G270). The RGO additives were dispersed in the epoxy matrix. Each sample was subjected to Integrated Current Transformer (ICT) electron beam at a constant dose of 100 kGy. The mechanical properties of Nano composites were tested by a Universal Testing System (UTS) and were correlated to the variation of their constituents' molecular structure obtained by Fourier Transformation Infrared (FTIR) spectroscopy, Dynamic Mechanical Analysis (DMA) and Electric Resistivity (ER). The results revealed an enhancement in the mechanical properties of epoxy matrix after the addition of RGO except for (RGO-33C) and preservation of the mechanical properties even after irradiation.

Keywords

Space environment; Electron beam; Charged particles; Carbon fiber; Epoxy; Composite materials

Introduction

Scientists and engineers have developed advanced materials for manned spacecraft and satellites for a range of sophisticated applications in space exploration, transportation, and communication. The materials used on the exterior surfaces of spacecraft are subjected to many environmental threats that can degrade the exterior materials and components [1]. One of these threats is charged particles (ionizing radiation). Space radiation is comprised of atoms in which electrons have been stripped away as the atom accelerated in interstellar space to speeds approaching the speed of light only the nucleus of the atom remains [2]. The Low Earth Orbit (LEO) is normally at an altitude of (200 Km-1000 km) above the Earth. LEO satellites travel at a speed around 7.8 km/sec [3].

In this paper, the mission design was selected to be a remote sensing satellite in an altitude of 600 km, orbit inclination 97.7° for a five years orbital life. According to the mission parameters the ion beam intensity was calculated to be a 100 KGy for the whole satellite life time.

Polymer nanocomposites combine the functionalities of polymer matrices, such as low cost, easy process-ability, with the unique features of the inorganic nanoparticles such as high aspect ratio, excellent toughness, and high strength, good electrical and thermal conductivities. In the past few years, polymer nanocomposites with enhanced optical, mechanical, electrical, thermal, and fire-retardant properties have been developed [4]. Epoxy resin is one of the most commonly used thermosetting materials in SC structure due to its excellent mechanical properties and chemical stability [5]. However, epoxy composites with variable engineering applications are often limited by their brittle nature, poor electrical and thermal properties. A simple solution to overcome this problem is to modify the matrix molecular structure or to add compatible materials [6].

In 2017, Nguyen et al. was studying the thermal and mechanical properties of epoxy polymer after exposure to different doses of electron beam irradiation. The effect of different EB doses (30 kGy, 100 kGy and 300 kGy) on thermal and mechanical properties was investigated. The characterization methods are Thermal Gravimetric Analysis (TGA), Universal Testing System (UTS), and Dynamic Mechanical Analysis (DMA). The main results revealed a slightly increase in the thermal properties of the epoxy polymer after irradiation. Also, the increase of the tensile strength and Young's modulus of the polymer by the EB action up to 100 kGy dose and then begins to decrease. The irradiation dose increased, the elongation decreased [7].

In 2019, Khan et al. studied the effect of Reduced Graphene Oxide (RGO) on the mechanical and thermal properties of epoxy nanocomposite. RGO were dispersed in ethanol by using bath sonicator and mixed with (0.1 wt. %, 0.2 wt. %, 0.3 wt. %, 0.4 wt. %, 0.5 wt. %) respectively in epoxy to obtain *in situ* composites. The XRD and SEM images confirmed intercalation, exfoliation and distribution patterns of RGO in epoxy. The mechanical properties increased with the increase of RGO up to 0.3 wt. % due to dispersion quality. The thermal properties showed insignificant improvement [8].

In 2017, Ahmed Alzahrany et al. investigating the effect of Reduced Graphene Oxide (RGO) on the structure and properties of epoxy. Epoxy-RGO nanocomposites were prepared by mixing epoxy with RGO in presence of a solvent, tetrahydrofuran. X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were performed to characterize the structure and morphology of the nanocomposite. The characteristic peaks for the functional groups of both RGO and polymer matrix were found in FTIR spectra of nanocomposite. RGO interlayer spacing increased in nanocomposite according to XRD results. SEM images clarified that there was a relatively good

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dispersion of graphene in epoxy matrix. The incorporation of 1.0 wt. % RGO into epoxy matrix raised the Young's modulus from 2.71 GPa to 3.56 GPa. It increased the degradation measured at 50% weight loss from 412°C to 433°C. The thermal conductivity was improved by up to 85%, reaching the maximum value of 0.36 Wm-1K-1 at 1.0 wt. % RGO. The same addition also significantly improved the electrical conductivity [9].

In this work, a Reduced Graphene Oxide (RGO) with various preparation methods was dispersed in an epoxy matrix to be used in space structure applications. Three types of RGO's were synthesized and investigated to show the effect of electron beam irradiation on thermal and mechanical properties. The properties were tested by using the Universal Testing System (UTS), Dynamic Mechanical Analysis (DMA), Fourier Transform Infra-Red (FTIR) and Electric Resistivity. All characterization procedures were applied to the samples prior and post Electron Beam (EB) irradiation [7].

Experimental Work

Material

The epoxy nanocomposites samples were made from three main constituents; epoxy matrix, carbon fiber reinforcement, and RGO nanoparticles as an additive for enhancing the nanocomposite properties.

First, the epoxy matrix (biresin®) was made by mixing (73%) of the resin part A (CR 82®) and the remaining percentage from the hardener part B (CR 80-6®). The additives are considered as a part of the resin weight percentage.

Second, three different types of RGOs were used in this research symbolled as (G-24N, G-33C, G-270). The first sample (G-24N) is N-doped RGO with 3D structure. Its atomic composition is listed below in Table 1. It was synthesized by thermal dissociation of PET waste bottles with urea at 800°C for 5 hours. The second sample (G-33C) was prepared from waste PET bottles which were crushed and sieved to obtain particles with size fraction (1 mm to 3 mm). Then (2 g) of raw PET waste was introduced into an enclosed 50 mL stainless-steel autoclave reactor (SS-316). The closed stainless steel reactor was placed inside the center of an electric furnace and heated to 800°C with a rate of 8°C/min and maintained for 1 hour. The resulted dark product was collected and crushed. The bottles waste was used as a precursor for graphene synthesis [10] the third sample (G-270) is an N-doped multilayered graphene nano-sheets. The graphene nano sheets were well exfoliated. The doped N had dominated pyloric conformation followed by pyridinic and lastly graphitic. It was created by hydrothermal treatment of glucose solution under mild synthetic conditions. All the prepared RGO compound size is about 30 μm diameter with density 0.95 g/cm3 (Table 1).

Table 1: Atomic composition of RGO compounds.

No.	Sample designation	Atomic composition %			
		Carbon	Oxygen	Nitrogen	Hydrogen
1	G-24N	83.3	13.9	2.8	
2	G-33C	90.49	7.379		2.131
3	G-270	89	7.2	3.8	

Third, Carbon fiber was used in the form of plain fabric made by "SELCOM company" product name "CBXS200®" with an areal density of 206 g/m2. The mechanical properties related to 6K with 50% carbon, 5 threads/cm the tensile strength 2250 MPa and 1.5% elongation.

Specimen preparation

Sonication process: The sonication process was executed by using "SONICS VCX750®" sonicator to prepare the sample of epoxy resin with RGO. The sample was stirred for 5 min at room temperature then it was mechanically stirred for about 60 min in the sonicator with 9 KHz frequency and 750 W power.

Sample preparation to nanocomposites sheets.

The nanocomposites were prepared by mixing 50 g of neat epoxy with 0.3 wt. % RGO and 27 wt. % hardener. The solution was reinforced by a carbon fiber plain forming a carbon fiber/epoxy sheet. Using a well waxed glass plate with a double faced tape border, a thin uniform layer of epoxy/nanoparticle mixture was rolled. A carbon fabric ply was then placed on the epoxy/nanoparticle layer. This process was repeated four times before a layer of foam was added to absorb the excessive resin used in the hand layup process. The whole structure was wrapped with vacuum bag, taped and subjected to the vacuum oven with 45°C for 24hrs.

Electron beam source: The prepared samples were irradiated at a constant dose of 100 kGy for the calculated dose related to the satellite mission orbit and lifetime. The exposure was applied in air using an ICT electron beam accelerator at the National Center for Radiation Technology, Cairo, Egypt. The irradiation was done at a beam current of 16 mA, energy of 2.7 MeV, and a conveyor speed of 1.08 m/min.

Evaluation method

Universal testing system: The mechanical properties of both the non-irradiated and irradiated tensile specimens were evaluated using the universal testing machine 810 Material Test System (MTS). All the tests were performed at room temperature (23°C ± 3°C) and relative humidity of (50% ± 10%) according to the test standard ASTM D3039.

Fourier Transfer Infra-Red (FTIR): Infrared spectroscopy was done by FTIR spectrometer JASCO 6600 by grinding the candidate sample and the ground up mixture was compressed between two KBr pellets. A total of 45 scans were taken for all of the composite samples; they were recorded at 4000 cm-1 to 400 cm-1 with a resolution of 4 cm-1 in transmittance mode. This process was used to study the variation in chemical structure before and after irradiation.

Dynamic Mechanical Analysis (DMA): It was conducted using Triton® Technology for dynamic mechanical analysis; the three point bending mode was used to determine Tan delta, storage and loss modulus of each sample. The sample dimension was (10 mm × 2.5 mm × 2.4 mm). The frequency used was 1 Hz and the temperature was 20°C -160°C, with a heating rate of 5°C/min.

Electric resistivity: The electrical resistances were measured by Keithley 2635A® System Source Meter at the National Center for Radiation Research and Technology, Cairo, Egypt. This test was subjected to recognize the dispersion quality of RGO in the epoxy matrix. Each sample dimension was (10 mm × 10 mm × 2.4 mm). The sample was subjected to a current of 1 mA and a voltage of 20 V. The device clamps were connected to positive and negative electrical current. The samples were placed between the clamps that contain a

subjected circular area of 12.57 mm2.

Results and Discussion

Tensile test

This test showed different behavior of mechanical properties of each sample. The tensile stress, elongation and young's modulus were determined for each sample prior and post irradiation. As shown in Figure 1, prior irradiation the tensile stress of neat epoxy was increased after adding RGO types except for G-33C. This shows that the addition of nanoparticles increases the epoxy mechanical properties (Figure 1) [6].

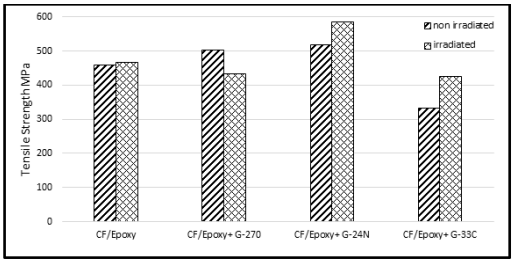

Figure 1: Tensile strength of nanocomposites irradiated and non-irradiated.

After irradiation, Epoxy/G-24N and Epoxy/G-33C showed an increase in tensile stress compared by the corresponding sample prior irradiation. The by Epoxy/G-33C was shown an increase of 30% than the non-irradiated sample. However, Epoxy/G-270 showed a decrease in its tensile stress. This is due to the atomic compositions where G-33C contains the highest carbon content forming a harder structure and the absence of nitrogen doping caused the decrease of electron mobility inside the low-lattice of graphene in comparison by the other RGO (10). On the other hand, for G-270 nitrogen doping was increased causing higher electron mobility inside the graphene low-lattice forming a new crystalline structure (Figure 2) [11].

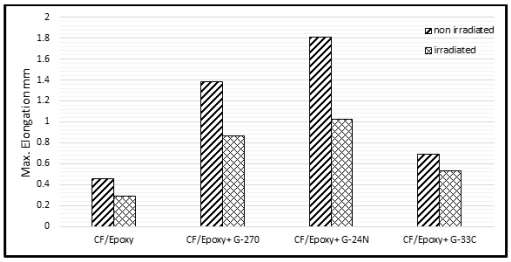

Figure 2: Elongation at peak of nanocomposites irradiated and non-irradiated.

Also, the elongation of the candidate samples was determined by the tensile test as shown in Figure 2. Prior irradiation, the elongation of neat epoxy was increased by adding nanoparticles. Herein, after electron beam irradiation, the elongation of neat epoxy was decreased by 37%. In addition, all nanocomposites samples exhibit a decrease in elongation. The highest decrease was shown in Epoxy/G-270 by 38% (Figure 3).

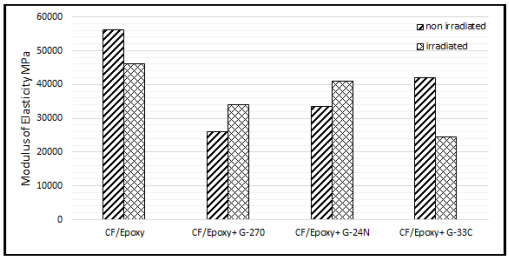

Figure 3: Modulus of elasticity of nanocomposites irradiated and non-irradiated.

Last parameter was young's modulus which is showed in Figure 3. Prior irradiation, the neat epoxy young's modulus decreased by adding RGO. The highest reduction value of Young's modulus was obtained by Epoxy/G-33C prior and post irradiation due to the highest increase in strength that caused by the new cross-links changing the mechanical behavior from ductile to slightly brittle nanocomposite [11]. Post irradiation, the young's modulus of neat epoxy decreased by 10% compared with non-irradiated neat epoxy. On the other hand, The Modulus of elasticity exhibits an enhancement of 30% and 12.4% for Epoxy/G-270 and Epoxy/G-24N nanocomposite respectively. So, the highest modulus was obtained by Epoxy/G-270 optimizing this new nanocomposite structure.

Fourier Transform Infrared (FTIR)

FTIR results of irradiated and non-irradiated samples show the wavelength shift indicates the dehydrogenation and the intensity variation represents the bond cleavage or formation [12]. The FTIR result showed no losses of functional groups after the addition of nanoparticles in the epoxy matrix. This is important because the reactive sites are conserved for future manipulation (Figure 4).

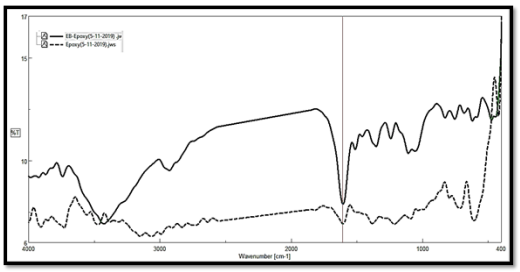

Figure 4: FTIR comparison between Neat Epoxy and Epoxy after Irradiation with EB.

In Figure 4, the comparison between the neat epoxy and the irradiated neat epoxy were studied to find the behavior of epoxy due to electron beam irradiation. From the plotted curve prior irradiation an increase in some peaks intensity were appeared due to dehydrogenation of the sample, on the other hand there is no change in peaks by diminish or creating new group indicating no change in the chemical structure of the epoxy due to electron beam.

The samples CF/Epoxy+G270 showed a new NH2 and N-H bonds were found for epoxy matrix, due to the addition of RGO, at peak of (1599.66 cm-1, 1596.77 cm-1). This is due to RGO doping by nitrogen. Additionally, the samples contain RGO-33C and RGO-24N, display no significant change in the chemical structure of the material after Adding RGO additive.

The samples CF/Epoxy+G270 showed a new NH2 and N-H bonds were found for epoxy matrix, due to irradiation at (3394.1 cm-1, 3429.78 cm-1) respectively, these peaks can be interpreted as the a normal behavior due to the dehydrogenation of the sample in the presence of nitrogen used in the doping of RGO and the effect of exposing the sample to EB in the presence of oxygen from air. The irradiation sample of CF/Epoxy+G24N shows a new peaks at 3284.18 cm-1 (OH) and 2971.77 cm-1 (C-H), these new peaks can be attributed to the opening of the epoxy ring by amine, forming a free hydroxyl group and breaking the original (C-O) of the epoxy (Figure 5).

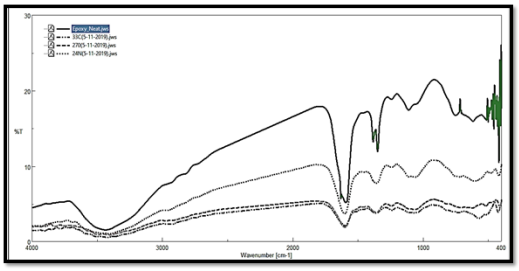

Figure 5: FTIR for all tested samples before irradiation.

The sample containing RGO-33C, displays no significant change in the chemical structure of the material after undergoing irradiation. All the functional group peaks were preserved with no statistical differences in the shifts. This means that the samples underwent a physical but not a chemical change (Figure 6).

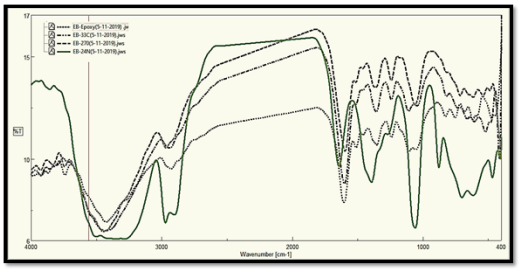

Figure 6: FTIR for all tested samples after irradiation.

Dynamic Mechanical Analysis (DMA)

It is an experimental method to measure the storage, loss modulus and damping factor. The storage and loss modulus are used to determine the glass transition temperature (Tg). The amount of damping indicates how much energy can be absorbed by the material (Figure 7).

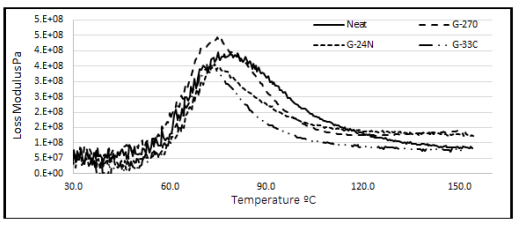

Figure 7: Loss modulus *vs.* temperature.

The loss modulus was obtained in Figure 7, the effect of adding RGO was compared with neat epoxy. There is no significant change noticed in loss modulus when adding RGO to epoxy, clarified that the mechanical properties of the sample does not depend on the additive type within various temperatures.

The storage modulus variation with temperature represented in Figure 8, the storage modulus decreased while adding RGO to epoxy, in temperature ranges from 20°C to 80°C, the RGO type did not affect the modulus while in the range 80°C and above RGO (G-270) shows the lowest value of storage modulus while G-24N and G-33C remains same as neat epoxy sample (Figure 8).

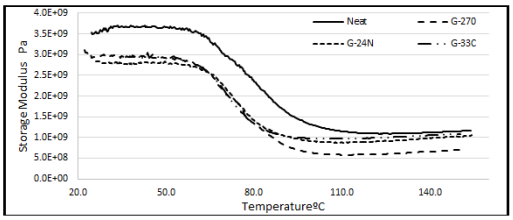

Figure 8: Storage modulus *vs.* temperature.

The glass transition temperature can be easily determined from the highest peak of damping factor shown in Figure 9, the highest Tg was recorded by neat epoxy at 93.17°C while adding additives reduce the Tg values to be 90°C, 87.76°C and 78.61°C for (G-270, G-24N and G-33C) respectively. Moreover, the smoothness in the graph indicates the well dispersion of RGO in epoxy matrix. (Figure 9).

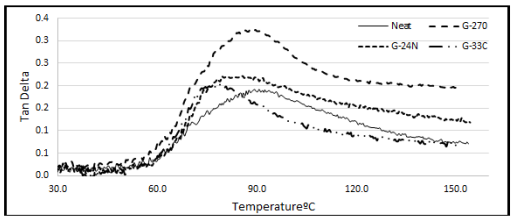

Figure 9: Tan delta *vs.* temperature.

Electric resistivity

The electrical resistivity is known as the resistance of the material towards the flow of electric current passing through it. This test was used to determine the electrical conductivity of each sample. Table 2 shows the electrical conductivity calculated by this equation: σ=L/(R*A).

Where (σ) is the electrical conductivity S/cm, (L) is the thickness in cm, (R) is the resistivity in ohms and (A) is the subjected area (0.125664 cm2).

Prior to irradiation, electrical conductivity of neat epoxy increased by adding RGO where the sample electric properties changes from an insulating material to a semi-conductive material (Table 2) [13].

Table 2: Electrical conductivity and resistivity.

	Resistivity Ω		Electrical conductivity S/cm	
	Before Ion beam	After Ion beam	Before Ion beam	After Ion beam
Neat	0.2×10^{9}	12933	8.7×10^{-9}	1.4×10^{-4}
G-270	669	333	3.1×10^{-3}	6.1×10^{-3}
G-24N	1047	211	1.8×10^{-3}	9.1×10^{-3}
G-33C	957	263	2.1×10^{-3}	7.9×10^{-3}

The reason behind this enhancement is the good reduction of graphene oxide because the electric conductivity is directly proportional with degree of GO reduction [14]. Also, FTIR spectroscopy clarifies the formation of new bonds such as N-H was formed at 3394.1 cm-1 and 3429.78 cm-1. These new bonds increased the flexibility of the epoxy and facilitate the movement of current inside the epoxy matrix.

Conclusion

The non-irradiated samples showed an enhancement in the mechanical properties after adding G-270 and G-24N. However, the irradiated samples showed an increase in the mechanical properties by adding all the additives type. The FTIR spectroscopy displayed every peak of epoxy and RGO with no significant change in the peak intensity after irradiation. This means there was no change in the chemical structure of Epoxy/nanoparticle nanocomposites.

Finally, the addition of nanoparticle enhanced the mechanical properties of non-irradiated CF/epoxy samples. Also, the electron beam irradiation did not cause a failure of the samples but it acted as more enhancement to the nanocomposite mechanical properties through the mission life time. The usage of the candidate materials are proper to the similar kind of space missions as a structure materials for SC. Especially CF/epoxy-24N; this Nano-composite was shown a improvement in the mechanical properties prior and post irradiation to figure that there is no problem in using it in space through the SC life time also, the conductivity shows an increase to be the most suitable material to be used in outer plates structure of the SC.

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