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Effect of Nanoparticles on the Carbon fiber/Epoxy composite mechanical properties

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Abstract. : The mechanical properties of the carbon fiber/Epoxy composite were studied after the addition of nanoparticles for space applications. Three different nano-particles (Al_2O_3, MWCNT, and RGO) were dispersed in the epoxy matrix. Each sample was subjected to ICT electron beam radiation at a constant dose of 100 kGy. The mechanical properties of nanocomposites were tested by a universal tensile testing machine and were correlated to the variation of their constituents' molecular structure obtained by the Fourier Transformation Infrared (FTIR) spectroscopy. The results revealed an enhancement in the mechanical properties of epoxy matrix after the addition of nanoparticles and preservation of the mechanical properties even after irradiation.

1. INTRODUCTION

Scientists and engineers have developed advanced materials for manned spacecraft and satellites for a range of sophisticated applications in space exploration, transportation, global positioning and communication. The materials used on the exterior surfaces of spacecraft are subjected to many environmental threats that can degrade exterior materials and components[1]. One of these threats is charged particles (ionizingradiation). 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 – eventually, only the nucleus of the atom remains[2]. The NASA limit for radiation exposure in low-Earth orbit is 0.50 Sv/year, or 50 rem/year[3].A low Earth orbit is normally at an altitude of less than 1000 km and could be as low as 160 km above the Earth. At low earth orbit satellites travel at a speed of around 7.8 km per second[4].

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 strength and other properties like electrical and thermal conductivities. In the past few years, polymer nanocomposites with enhanced optical, mechanical, electrical, thermal, and fire-retardant properties have been developed[5]. Epoxy resin is one of the most commonly used thermosetting materials in spacecraft due to its excellent mechanical and electrical properties and chemical stability[6]. However, epoxy materials with varying engineering applications are often limited by their brittle nature and poor electrical, thermal properties. A simple solution to overcome this problem is to modify the matrix molecular structure or to add compatible materials[7].

In 2017, A. T. Nguyen*et. al*. studied the thermal and mechanical properties of epoxy polymer after exposure to different doses of electron beam irradiation. Different irradiation doses of 30, 100 and 300 kGy were used. The effects of those doses on thermal and mechanical properties of the epoxy polymer were then investigated by the methods of thermal gravimetric analysis, tensile test, and dynamic mechanical analysis. The main results revealed a slightly increase in the thermal properties of the epoxy polymer after irradiation at the heating in air. Also the increase of the tensile strength and Young's modulus of the epoxy polymer by the action of electron beam up to dose of 100 kGy and then it begins to decrease at higher doses. As the irradiation dose increased,the elongation at break decreased[8].

In another reportin 2017, A. T. Nguyen*et.al*.examined the effect of electron beam irradiation on the thermal and mechanical properties of the epoxy composites filled with aluminum nanoparticles at percentage of 0.35 wt% was tested. Different range of doses of 30, 100 and 300 kGy were used. The methods of thermal gravimetric analysis, tensile test, and dynamic mechanical analysis were used to investigate the effects of doses on thermal and mechanical properties of the aluminum-based epoxy composites. The results showed that the epoxy/Al_2O_3 composites showed good irradiation resistance. The enhancement of the thermal and mechanical properties of the aluminum-based epoxy composites were sustained until the irradiation dose of 100 kGy then it begins to decrease at higher doses[9].

G. Szebényi *et.al*.investigated the effect of electron irradiation on the mechanical properties of multiwall carbon nanotubes (MWCNT)/carbon fiber reinforced hybrid nanocomposites in 2010. In this article, carbon fiber/epoxy composite and MWCNT/carbon fiber/epoxy hybrid nanocomposite laminates had been irradiated using a high energy electron gun with multiple doses. The effect of the electron irradiation had been characterized using three-point bending, interlaminar shear and instrumented falling weight impact tests. The main two hypothesis of this study were that the use of the electron beam irradiation on the specimens can excite the electrons of the carbon nanotubes and the matrix to create free radicals in the system. Where after recombination these free radicals could form new covalent bonds between the nanotubes and the matrix. These new bonds will improve the nano-sized reinforcing materials and the matrix. The second hypothesis is that the effect of electron irradiation could change crosslinking density and chainlength of the matrix[10].

In this work , aluminum oxide (Al_2O_3), multi-wall carbon nanotube (MWCNT) and reduced graphene oxide (RGO) were dispersed in an epoxy matrix for space applications. These three nanoparticles were chosen because they showed enhanced thermal and mechanical properties[8][9][10].

2. EXPERIMENTAL WORK

2.1Material

Epoxy nanocomposites samples were made using biresin two parts matrix; part A CR82 (resin) and part B CR80-6 (hardener). Three different nanoparticle materials were added to the epoxy matrix.

Table 1: summarize different types and characteristics of each nanoparticle used in this research.

Nanoparticles	Characteristics
Aluminium oxide (Al_2O_3)	White powder of Gamma- Al_2O_3, with purity of 99.99% and a 0.4 to 1.5 nanometer diameter
Multi-walled carbon nanotubes (MWCNT)	MWCNT’s were produced by a high-yield catalytic process based on chemical vapor deposition (CVD) with an outer mean diameter of (8-10 nm) and inner mean diameter (4nm) and length from (5-10 μm). The purity of neat MWCNTs was greater than 90%.
Reduced graphene oxide (RGO) Three different type of reduced graphene oxide were used in this thesis (24N, 33C, G270).	1. Sample G24N also called 4NG: It is N-doped reduced graphene oxide with 3D structure. Its atomic composition is 83.3 % C, 13.9 % O and 2.8 % N. It is synthesized by thermal dissociation of PET waste bottles with urea at 800˚C for 5 hr. 2. Sample G33C was prepared as sample 9C[11]but not in the same batch. Its atomic composition is 90.49% C, 2.131% H and 7.379% O. 3. Sample G270: It is an N-doped multilayered graphene Nano sheets. Its atomic composition is 89 % C, 7.2 % O and 3.8 % N. The graphene Nano sheets are well exfoliated. The doped N has dominated pyloric conformation followed by pyrdinic and lastly graphitic. It is synthesized by hydrothermal treatment of glucose solution under mild synthesis conditions.

2.2Specimen Preparation

2.2.1 Sonication process

Epoxy resin (50g) was mixed with the three different types of nanoparticles (Al_2O_3, RGO and MWCNT) at different weight percent (1%, 0.3%and 0.5% respectively) in epoxy resin by stirring for 5 minutes at room temperature. Then the nanoparticles were well dispersed in epoxy resin by using SONICS VCX750 sonicator for a constant time (1 h) with 9 KHz frequency and 750W power.

2.2.2 Production of nanocomposites sheet

Firstly,about 27wt. % of carbon hardener was added to the mixture of 50g epoxy and nanoparticles. Then using awell waxed glass sheet with a double faced tape border;the epoxy/nanoparticle mixture was rolled into a uniform layer. A carbon fiber fabric layer was then rolled on the epoxy/ nanoparticle layer. Followed by a second layer of the epoxy/nanoparticle mixture. This process was repeated two times then a layer of foam was added to absorb the excessive resin used in the hand layup process. The whole glass was wrapped with vacuum bag and taped with double tap. Finally, the whole sheet was vacuumed with -1bar at 45℃ for 24h.

2.2.3Radiation source

The samples were irradiated at a constant dose of 100 kGy in air using an ICT electron beam accelerator at the National Center for Radiation Research and Technology, Cairo, Egypt. The irradiation was done at a beam current of 16 mA, an accelerator energy of 2.7 MeV, and a conveyor speed of 1.08 m/min.

2.2.4 Evaluation method

The mechanical properties of both the non-irradiated and irradiated tensile specimens were evaluated using the universal tensile testing machine 810 Material Test System (MTS).All the tests have been performed at room temperature(23±3°C) and relative humidity of (50±10 %) according to the ASTM D3039. To explain and relate the variation of the mechanical properties of the irradiated material to the chemical structure, a Fourier Transform Infra-Red (FTIR) spectrometer JASCO 6600, shown in Fig. 1, was used.

Fig 1Fourier Transform Infrared Spectrometer FTIR JASC

3.RESULTS AND DISCUSSION

3.1 Tensile Test

Tensile test showed different behavior of mechanical properties of each sample. The tensile stress, elongation and young's modulus were determined for each epoxy/ nanocomposites before and after radiation as shown in figure (2). The tensile stress of Neat epoxy before irradiation was increased after the addition of nanoparticle for all additives except for G33C. This means that the addition of nanoparticles increases the mechanical properties of epoxy matrix [7].

After irradiation, Epoxy/MWCNT, Epoxy/G24N and Epoxy/G33C nanocomposites showed an increase in their tensile stress. The highest tensile stress was obtained by Epoxy/G33C of 30%. However, Epoxy/G270 showed a slight decrease in their tensile stress.

This is due to their atomic compositions where G33C contains the highest carbon content forming a harder structure and no nitrogen doping causing lower electron mobility inside the low-lattice of graphene in comparison with the two other reduced grapheneoxides (RGO)[11]. On the other hand, for G270 nitrogen doping was increased causing higher electron mobility inside the graphene low-lattice forming a new crystalline structure[12].

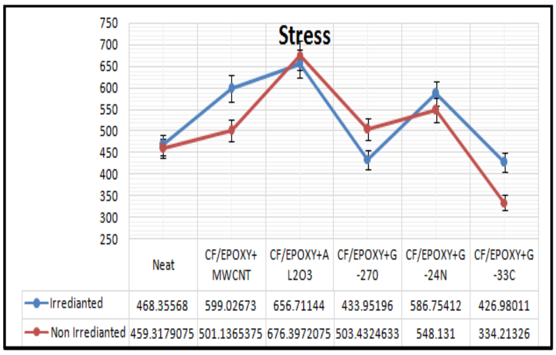

Fig 2 Tensile stress of neat epoxy and epoxy nanocomposites irradiated and non- irradiated

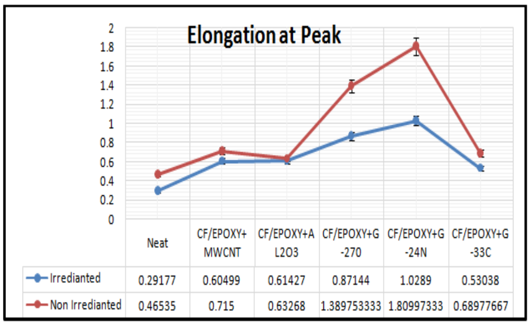

Fig 3 Elongation at peak of neat epoxy and epoxy nanocomposites irradiated and non-irradiated

Also, the elongation of neat epoxy and epoxy nanocomposite was determined by the tensile test as shown in figure (3). Before irradiation, the elongation of neat epoxy was increased after the addition of nanoparticles. After electron beam irradiation, the elongation of neat epoxy was decreased by37%.

Also, all nanocomposites samples exhibit a decrease inelongation at peak.The highest decrease was achieved in Epoxy/G270 by 38%. As mentioned before, the addition of nitrogen doping increases electron mobility in graphene low- lattice forming a new crystalline structure.

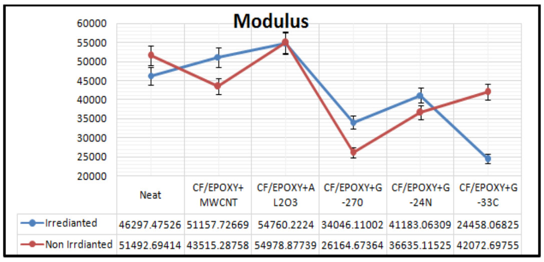

Fig 4 modulus of elasticity of neat epoxy and epoxy nanocomposites irradiated and non-irradiated

The last mechanical property obtained from the tensile test was the young's modulus as shown in figure (4). Before electron beam irradiation, the neat epoxy young's modulus decreased after the addition of nanoparticles except for Al_2O_3. The highest decrease was obtained by Epoxy/G33C before and after irradiation this due to the highest increase in strength caused by the new cross-linked formed inside the nanocomposite structure changing the mechanical behavior from ductile to a slightly brittle nanocomposite[12]. After irradiation, the young's modulus of neat epoxy decreased by 10% less compared to non-irradiated neat epoxy. On the other hand, the young's modulus of Epoxy/Al_2O_3 remained constant even after irradiation[9]. However, The Modulus of elasticity exhibits an enhancement of 17.56%, 30% and 12.4% for Epoxy/MWCNT, Epoxy/G270 and Epoxy/G24N nanocomposite respectively. As mentioned, the highest modulus was obtained by Epoxy/G270 optimizing these new nanocomposite structure.

3.2 Fourier Transform Infrared (FTIR):

FTIR results of irradiated and non-irradiated epoxy/nanoparticles nanocomposites are shown in Table 2. FTIR consists of two main parameters wave number and absorption intensity variation (abs). The wavelength shift indicates the dehydrogenation(hydrogen loss) and the intensity variation represents the bond cleavage or new one formation[13].

Table 2: summarizing the results of FTIR for epoxy matrix and epoxy nanocomposites before and after irradiation

Bond	Wavelength (cm^{-1})[14],[15],[16]	Samples	Wavelength non irradiated	Wavelength irradiated
Finger print	~<1000	Epoxy	421.37 608.431 - 715.461 -	473.439 620.002 682.677 754.995 828.277
		Al2O3	419.442 529.364 603.61 -	434.869 554.434 619.038 695.212
		MWCNT	442.583 485.009 536.114 608.431 750.174 921.807	467.653 - 539.971 - 721.247 -

		G33C	410.763 574.683 727.996	415.585 522.615 607.467
		G24N G270	419.442 483.081 613.252 718.354 - 424.263 520.686 622.895 -	407.871 468.617 616.145 702.926 878.417 404.978 539.971 615.181 715.461
C=C-H	1607- 1510	Epoxy Al2O3 MWCNT G33C G24N G270	1605.45 - 1598.7 1594.84 1598.7 - -	1512.88 1605.45 1601.59 1605.45 1601.59 - -
NH_2	1590-1650	G24N G270	1601.59 1599.66	1643.05 1596.77
CH2-CH2	1450-1470	Epoxy MWCNT	- -1457.92	1459.85 -1511.92
CH2-CH3	1360-1390	Epoxy Al2O3 MWCNT G33C G24N G270	- 1359.57 1378.85 1359.57 1357.64 1357.64	1353.78 1355.71 1362.46 1357.64 1394.28 1364.39
Bond	Wavelength (cm^{-1})[14],[15],[16]	Samples	Wavelength non irradiated	Wavelength irradiated
sp^2 C-O	1200	Epoxy Al2O3 MWCNT G33C G24N G270	1215.9 1241.93 1253.5 1240.97 1242.9 1242.9	1241.93 1241.93 1243.86 1242.9 - 1242.9
sp^3 C-O	1025-1200	Epoxy Al2O3 MWCNT G33C G24N G270	- 1106.94 1040.41 1077.05 1107.9 1061.62 1060.66	1108.87 1110.8 1091.51 - 1111.76 1059.69 1062.59
Sp^3 C-H	2850-3000	Epoxy Al2O3 MWCNT G33C G24N G270	- 2929.34 2937.06 - -	2927.41 2940.91 2938.98 2939.95 2971.77 2966.95
Sp^2 C-H	3000-3100	Epoxy	3151.11	3426.89
N-H	3350-3500	G24N	3406.64	-
O-H	3200-3900	Epoxy Al2O3	- 3728.69 3428.81 3765.33	3426.89 3741.23 3411.46 3856.93

		MWCNT G33C G24N G270	3895.5 3417.24 3458.71 3855.01 3938.89 3423.99 3781.72 3907.07 - 3394.1 - 3824.15 - -	3963.96 3460.63 - 3861.76 - 3448.1 - 3928.29 3284.18 3857.9 3429.78 3743.15 3833.79 3927.32

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 conserve for future manipulation. The nanocomposites samples that contains G24N and G270 showed the existence of N-H and NH2 bonds due to the doped nitrogen to reduce graphene. Also, the result revealed that there was no significant change in peak intensity or peak position after irradiation.This means that there was a physical change not a chemical change in the epoxy/nanoparticle nanocomposites

4.Conclusion

In this article, the effect of Nanoparticles on the Carbon fiber/Epoxy composite mechanical properties was investigated by tensile test and characterize by Fourier Transform Infrared (FTIR) and tensile stress test.

Before irradiation, the tensile test showed an enhancement in the mechanicalproperties of Neat epoxy after the addition of nanoparticles for all additives exceptfor G33C. However,afterirradiation, the mechanical properties of neat epoxy and Epoxy / nanoparticle nanocomposites were increased for all additives even for Epoxy/G33C.

The FTIR result showed no losses of functional groups after the addition of nanoparticles in the epoxy matrix and there was no significant change in peak intensity or peak position after irradiation. This means that there was a physical change not a chemical change in the Epoxy/nanoparticle nanocomposites.

This means that the addition of nanoparticleenhanced the mechanical properties of epoxy matrix. Also, the electron beam radiation did not cause the failure of the epoxy nanocomposites but it acted as an assistant that enhanced the mechanical properties.

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