



## Effect of gamma and electron beam irradiation on PAN-carbon fiber composite

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### ABSTRACT

The aim of this study was to evaluate the effects of irradiation on structural, mechanical and thermal properties of PAN-carbon fiber composite. The overall applied doses were 250, 500, 750, and 1000 kGy. Irradiated and non-irradiated samples were characterized by scanning electron microscopy, Fourier transform infrared spectroscopy and differential scanning calorimetry. Tensile strength test was conducted to measure mechanical properties. Scanning electron microscopy was used to evaluate microstructural behaviors. Thermal behavior of the samples was studied by thermogravimetric analysis and differential scanning calorimetry. The results showed that by increasing gamma and electron doses the thermal behavior of the composites indicated a higher decomposition degree as a function of the temperature. Electron irradiated carbon fiber surfaces are relatively smoother than virgin fibers. Bulges after gamma treatment were decreased and surfaces were unrough.

*Keywords: composite, irradiation, SEM.*

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### 1. INTRODUCTION

Composite material is a system composed of two or more macro constituents differing in form and/or composition that are essentially immiscible in each other [1, 2]. Typically, the composite material consists of a binder or a matrix that surrounds and holds reinforcements at a place. The

separate characteristics of the matrix and the reinforcements contribute synergistically to overall properties of the composite material [3-5]. The interface between the fiber and the matrix is an important aspect in control of the overall properties of the composite [6].

High energy particle and photon irradiation have been investigated to improve the mechanical properties of carbon fiber (CF) materials, such as yarns, by introducing direct heteroatom or polymer cross-links between CF shells and tubes [7]. Someone think increasing the mechanical properties may be attributed to an increase in the frictional strength by radiation damage defects [8]. The mechanical properties of carbon fibers depend on the properties of the CF and the matrix, particularly on the effectiveness of the interfacial adhesion between the CF and the matrix. An irradiation can induce chemical and grafting reactions at any temperature in the solid, liquid and gas phases without any catalyst [9, 10]. In addition, gamma ray penetrates highly in various objects and can lead to a uniform distribution of radical initiating sites through the thickness of the irradiated samples without considering their shape and volume [9]; the method is not harmful for the environment and the risk of pollution is reasonably low [10].

Carbon fiber–reinforced epoxy composites exhibit high specific strength, high specific stiffness and good fatigue tolerance, which have led to numerous advanced applications [11]. Various surface treatment methods such as electrochemical, chemical, thermal, plasma discharge, rare earth solution and gamma irradiation have been employed to improve the adhesion between the CFs and various matrices. Irradiation changes the interfacial microstructures to improve the fiber–matrix interfacial bonding. Surface roughening and polar functionality are the factors that contribute to the interfacial adhesion in gamma-irradiated fibers [6]. In recent years, gamma ray and electron irradiation have been extensively investigated as means of altering properties of polymeric materials, such as films, fibers, powders and molded objects [12].

In this study, polyacrylonitrile (PAN)-based carbon fibers were irradiated by gamma ray and electron beam. Electron and gamma irradiations were combined to investigate their influences on the CF composites. Fourier transform infrared (FTIR) spectroscopy, SEM and thermogravimetric (TG) analysis were conducted and then the densities of irradiated fibers were determined to study

how the structure of fibers changes. Moreover, the effects of gamma ray and electron beam irradiation on the tensile strength of CFs was also investigated.

## 2. MATERIALS AND METHODS

A combination of mechanical and thermal testing, and microstructural analysis were used to determine the effect of gamma and electron irradiation on the candidate material. The PAN-based carbon fiber composite was investigated in the current study and constructed in cylindrical shape. The cylinder was cut into several segments with dimensions 15 cm x 15 cm x 2 cm. The segments were prepared for shear the strength standard.

Ring samples of CF composites were placed on a frame and uniformly irradiated by two high doses of electron beams (250 and 500 kGy) at the room temperature with the rate 4.54 kGy/h. Then the samples were deposited into the  $^{60}\text{Co}$  point-source irradiators and irradiated in two dose levels (250 and 500 kGy) at room temperature.

Untreated CF composite rings were tested in tensions to study the effect of the treatment on the tensile strength. The test was performed by Zwick 250 Universal testing machine with the speed of 0.5 mm/s at the room temperature.

In order to investigate the possible changes in the chemical composition of CFs by gamma and electron beam treatments, FTIR analysis was done by Bruker Perkin Elmer SPECTRUM BX in mid infrared range ( $4000\text{--}700\text{ cm}^{-1}$ ) on composite segments of untreated, 0.25 and 1.0 MGy samples to provide a 'fingerprint' of polymeric materials.

TG curves were obtained using a STA1500 Rheometric Scientific. About 10 mg of the sample were set at an alumina crucible and heated from  $25^{\circ}\text{C}$  to  $700^{\circ}\text{C}$  with a heating rate of  $10^{\circ}\text{C min}^{-1}$  under dynamic nitrogen atmosphere ( $50\text{ mL min}^{-1}$ ).

End faces of the tested shear specimens (5 mm width and 1 mm length) were prepared metallographically. SEM studies on untreated, gamma and electron beam treated composites were

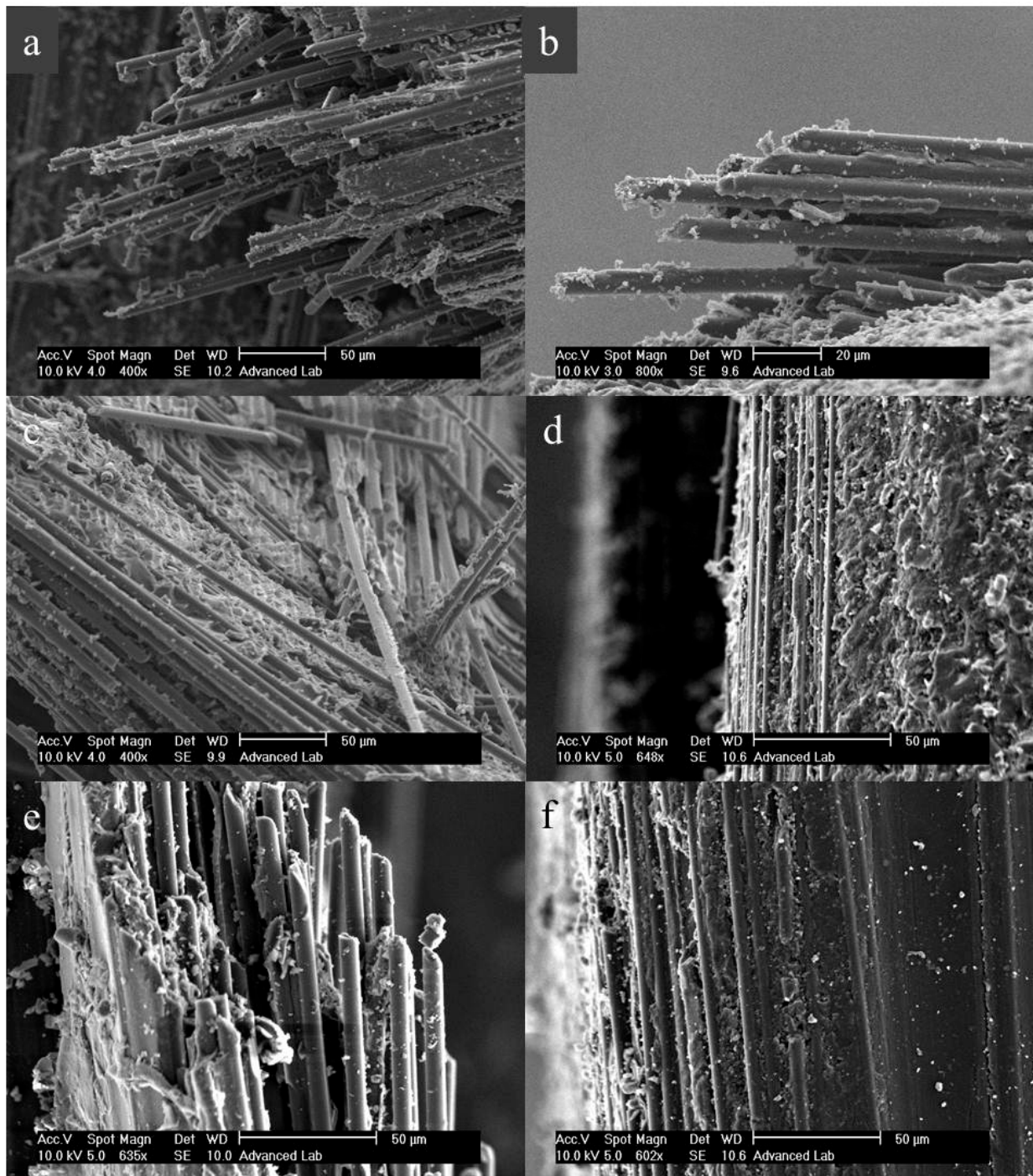
performed after gold sputtering via Zeiss EVO MA10 microscope to characterize the topographical changes on the fiber composite surfaces.

### 3. RESULTS AND DISCUSSIONS

#### 3.1 Scanning electron microscopy

Figure 1 shows the scanning electron micrographs of the fracture surfaces of untreated, gamma and electron beam treated fiber composites. Electron and gamma irradiation can cause two changes in the structure and physics of composite materials and polymers: these radiations may cross-linked bonds between molecular chains or breaking polymer chains. This can affect the chemical-physical and mechanical structure of materials. The untreated composites exhibited significant amounts of interfacial failures and their surfaces are rough (Figure 1, panels (a) and (b)). As electron irradiation was increased to 0.5 MGy, the surface of fibers was smoother than untreated ones as can be; see Figure 1 (c). By exposing the fibers to 250 kGy gamma dose, aggregated dose have been 750 kGy, the surface of fibers was smoother than prior cases (shown in Figure 1 (d)). At radiation dose of 1.0 MGy, shown in Figure 1 (e) and (f), the surface was unrough and some failures were observed. It suggested that the action of gamma and electron beam irradiation on composites promoted degradation of polymer chains. This means that the samples are more fragile and their strength was decreased. Fragility is due to the cumulative dose of gamma and electron beam that causes a change in the strength of irradiated samples. Therefore, cumulative dose broke the bonds in the samples and decreased the tensile strength.

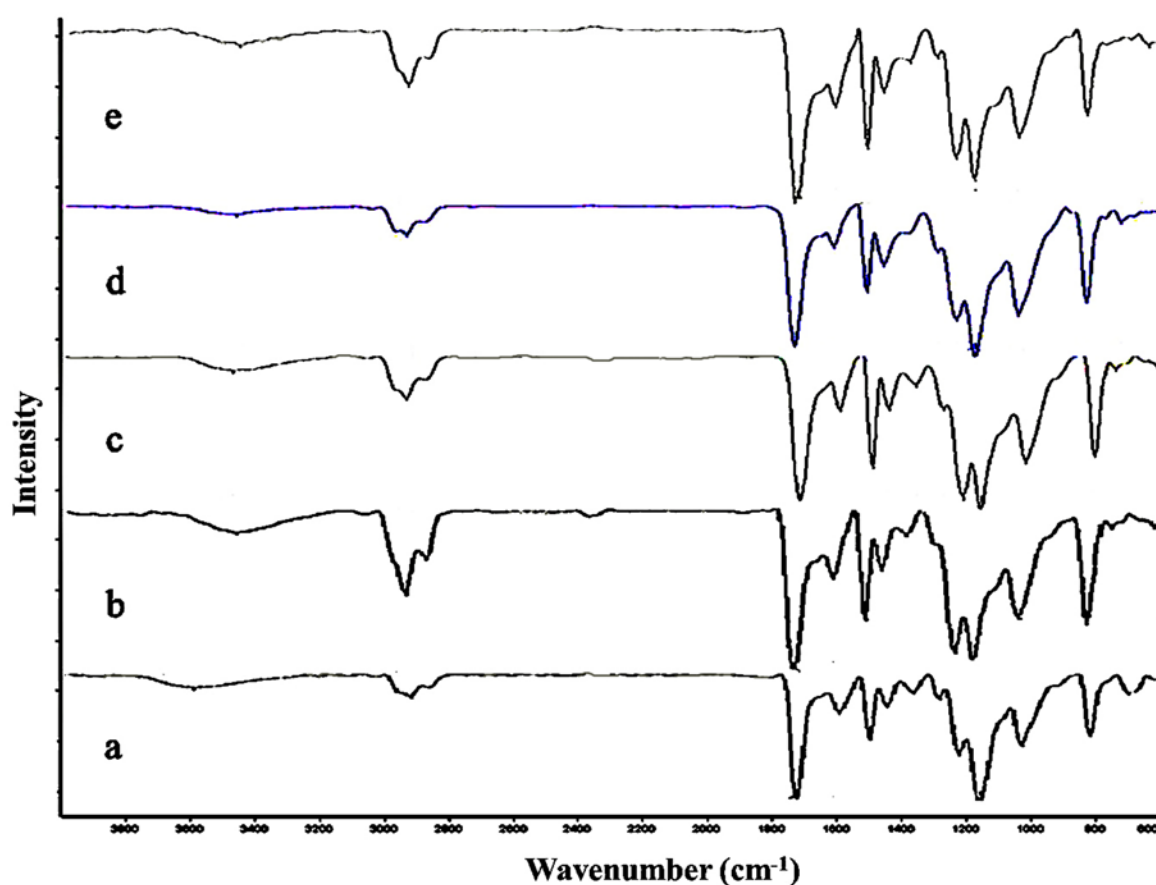
**Figure 1:** SEM micrographs of (a) non-Irradiated, (b) 250 kGy, (c) 500 kGy, (d) 750 kGy and (e, f) 1 MGy carbon fibers. Some of fractures are present within the resin parallel to the length of the fibers (f).



### 3.2 FTIR spectroscopy

FTIR spectra of untreated, gamma and electron beam treated CF are shown in Figure 2. The FTIR spectrum contained peaks of carbonyl, N-H, -C-CH<sub>2</sub>, C-N groups. According to Figure 2, the spectra did not exhibit significant changes, so that irradiation up to 1 MGy did not change CF composites structure.

**Figure 2:** FTIR spectra of carbon fiber composites in the frequency range of 3800 to 600 cm<sup>-1</sup>: (a) non-irradiated, (b) 250 kGy, (c) 500 kGy, (d) 750 kGy and (e) 1 MGy.

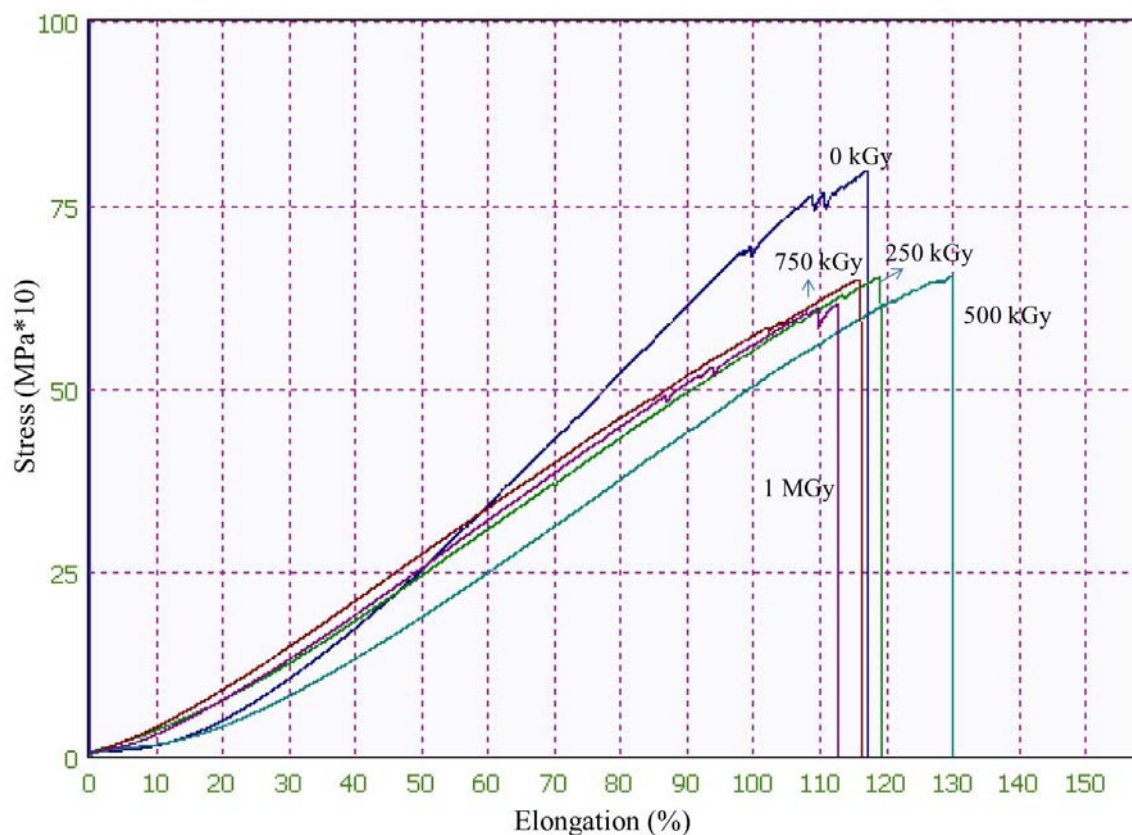


### 3.3 Tensile test

The effect of irradiation dose on tensile strength of carbon fiber rings is presented in Figure 3 and the results are summarized in Table 1. Tensile testing was conducted to determine the effects of gamma and electron beam irradiation on mechanical properties. A comparison of the tensile

properties of the samples exposed to the various radiation doses showed minimal differences in tensile behavior. Non-irradiated and 250 kGy electron irradiated samples showed a decreasing trend of stress/strain curve. While electron beam irradiation increased, remarkable changes were not observed. For gamma irradiation, minimal degradation on stress/strain has been shown. As observed, aggregate gamma and electron beam irradiation degrades the mechanical properties of the carbon fibers. Hoffman and Skidmore studied Radiation effects on epoxy/carbon-fiber composites. They demonstrated that stress/strain curves from samples exposed to high radiation doses do show erratic behavior, particularly at strains of 0.015 and higher. This suggested that the failure mechanism is different for highly irradiated samples and gamma radiation doses up to 2.0 MGy do not affect the carbon fibers significantly [13].

**Figure 3:** Stress/strain curves of representative of composite rings irradiated to doses of 0, 250, 750, 500 and 1000 kGy.



Irradiation induced the degradation of polymer chains: breaking its molecular structure and consequently reducing the tensile strength of CFs. The elongation of samples was increased between non-irradiated and 500 kGy dose and was degraded by more irradiation.

**Table 1:** Tensile properties data for carbon fiber composites irradiated to doses of 0, 250, 750, 500 and 1000 kGy.

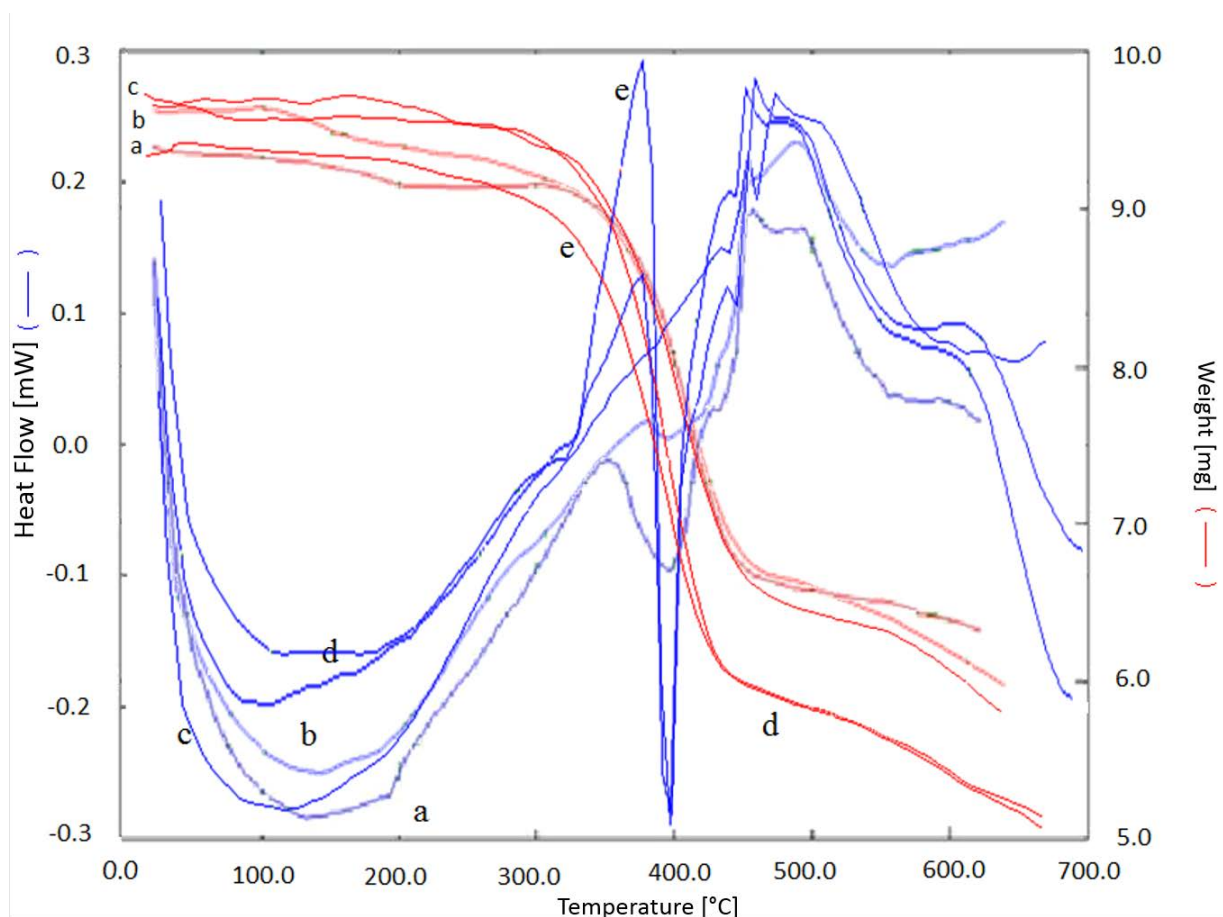
Sample	Strain (MPa)
Non- irradiated	798
250 kGy	653
500 kGy	654
750 kGy	632
1000 kGy	616

### 3.4 TG and DSC

Figure 4 shows representatives of TG and DSC curves for the studied samples. Here, it can be observed that by dose increasing no change occurred. However, DSC curves show that by increasing the applied dose the calefactory peak increased significantly due to sample decomposition at temperature of 381°C. This in 1000 kGy reach to maximum that indicating a higher decomposition degree as a function of temperature for irradiated samples. Hence, the glass transition temperature decreased from 198°C to 100°C for non-irradiated and 1000 kGy irradiated samples because the applied dose induced degradation polymer chains. In 2007, Giovedi and his co-workers investigated the effect of the thermal behavior of electron beams on CF composites. They obtained that the values of glass transition temperature decreased from 92°C for non-irradiated sample to 85°C for sample irradiated with 1000 kGy [14].



**Figure 4:** DSC ( blue) and TG (red) curves of carbon fiber composite in N<sub>2</sub> atmosphere for: (a) non-irradiated, (b) 250 kGy, (c) 500 kGy, (d) 750 kGy and (e) 1 MGy samples.



#### 4. CONCLUSION

The effect of gamma ray and electron beam irradiation on the structure and mechanical properties of CF composites were characterized by microstructural analysis. From these limited results, this composite appeared to be resistant to a dose of 250 kGy and it is similar to non-irradiated material. The thermal behavior, observed for the studied samples, exhibited that for doses higher than 500 kGy the changes occurred. The TG analysis revealed no substantial variation during gamma and electron beam irradiation. Degradation of the TG curve between 300-430°C was be-

cause of a decrease in the epoxy network properties. The damage threshold dose for the CF composites is unknown but the resin appeared to be slightly degraded at a dose of 0.5 MGy, with more changes at higher doses. Electron irradiation relatively smoothed the surface of CFs in comparison to non-irradiated fibers. After gamma exposure, the roughness of the surfaces decreased.

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