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Evaluation of the mechanical properties of carbon fiber after electron beam irradiation

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Abstract

Carbon fibers are used as reinforcement material in epoxy matrix in advanced composites. An important aspect of the mechanical properties of composites is associated to the adhesion between the surface of the carbon fiber and the epoxy matrix. This paper aimed to the evaluation of the effects of EB irradiation on the tensile properties of two different carbon fibers prepared as resin-impregnated specimens. The fibers were EB irradiated before the preparation of the resin-impregnated specimens for mechanical tests. Observations of the specimens after breakage have shown that EB irradiation promoted significant changes in the failure mode. Furthermore, the tensile strength data obtained for resin-impregnated specimens prepared with carbons fibers previously irradiated presented a slight tendency to be higher than those obtained from non-irradiated carbon fibers.

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Keywords: Carbon fiber; EB irradiation; Composites; Tensile strength

1. Introduction

Composite materials are systems composed of two or more constituents differing in form and/or material composition that are essentially immiscible in each other [1]. Polymeric composites are made of a polymeric matrix (resin) and a reinforcement material (fibers). Important factors for its performance are: orientation, length, shape and composition of the fiber, mechanical properties of each component and fiber–matrix adhesion [2]. Therefore, the behavior of a carbon fiber/epoxy

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matrix depends on the adhesion between these components, which have different chemical structure. It is also important to consider the sizing material on the fiber surface, which plays an important role in the adhesion process.

Electron beam (EB) radiation processing is being used for cross-linking of such composite matrices [3,4]. Main advantages of this process are: low temperatures, fast reaction time, low emission of volatile materials and a product with improved mechanical properties. The action of EB radiation on polymeric materials promotes mainly two processes: (a) cross-linking, that is the formation of chemical links between molecular chains, and (b) degradation or scission of polymer chains, which destroys its molecular structure. These chemical transformations result in changes in the physical and mechanical properties of the polymers. Although these effects occur simultaneously, one plays a dominating role depending on the chemical structure of the polymer, radiation dose and overall experimental conditions. Cross-linking improves mechanical properties whereas scission leads to a deterioration of the irradiated material. In addition, EB radiation also promotes excitation reactions on the fiber/matrix interface resulting in improved adhesion property [5].

The aim of this paper was to evaluate the effects of electron beam radiation on the tensile properties of two different carbon fibers used for structural applications containing different sizing material and number of filaments per roving. For this propose, the experiments were carried out using resin-impregnated specimens prepared using carbon fibers previously irradiated.

2. Experimental

2.1. Samples

Two different commercial carbon fibers roving of high tensile strength were studied. One carbon fiber roving contained 6000 filaments (6k) and the other 12000 (12k), each one had a different sizing material. The content of sizing material on both carbon fibers was of about 1.5 wt%.

2.2. Fourier transform infrared analysis (FTIR)

Identification of the sizing materials was carried out by FTIR analysis. Sizing was removed from the carbon fiber surface by dissolution in acetone at room temperature. For this procedure, it was used three meters of fiber and about 50 mL of acetone. Fibers and solvent were left in contact for about five hours and after this period, the acetone was evaporated from the solution at room temperature and the residue obtained was analyzed in an FTIR equipment model Nexus (Nicolet) from 4000 to 400 cm^{-1}.

2.3. EB irradiations

EB irradiations were carried out at the IPEN-CTR facilities using a 1.5 MeV and 37.5 kW Dynamitron Electron Accelerator model JOB-188. Irradiation conditions were: energy 0.555 MeV, electron-current 6.43 mA and dose rate 44.81 kGy s^{-1} to reach overall doses of 50, 100, 200 and 300 kGy.

2.4. Mechanical tests

Tensile properties of the fibers were determined as resin-impregnated specimens thermal cured according to ASTM D4018 [6]. The specimens for testing were prepared after EB irradiation of the carbon fibers. The resin formulation for the impregnation was a conventional epoxy for thermal cure at a maximum temperature of 130 °C for 8 h. Tensile measurements were carried out in an Instron Universal testing machine model 4206 with an extensometer in accordance to ASTM E 83 [7]. Experimental data obtained for each type of fiber were load and elongation at breakage. Volumetric fiber densities of 6k and 12k roving had been previously determined by a liquid displacement technique [8] and, from these values, it was calculated the fibers linear densities. From the load and elongation results and linear and volumetric density values, tensile strength (TS) and Young's modulus (E) were calculated for the test specimens. A set of resin-impregnated specimens of each carbon fiber roving as received (without having been EB irradiated) was prepared and used as blank.

Mechanical data were evaluated after measurements of six specimens.

2.5. Scanning electron microscopy (SEM)

SEM micrographs of the fiber surfaces from fractured samples were obtained using a scanning electron microscope model JXA-6400 (JEOL). The samples were not coated with a conducting material prior to examination.

3. Results and discussion

The volumetric densities for 6k and 12k carbon fibers were (1.76 ± 0.02) and $(1.77 \pm 0.02)\ g\,cm^{-3}$ and their linear densities, (0.407 ± 0.002) and $(0.764 \pm 0.002)\ g\,m^{-1}$, respectively.

The FTIR spectra showed that the sizing materials were different for each of the carbon fiber. For 6k carbon fiber the main absorption bands and their probable attributions were: 3434–3403 cm^{-1}, νOH; 3057–3036 cm^{-1}, νCH aromatic; 2965, 2929 and 2872 cm^{-1}, νCH_3 and CH_2; 1736, 1711–1709 cm^{-1}, νC=O; 1608, 1581 and 1512 cm^{-1}, νC=C aromatic; 1460 cm^{-1}, δCH_3 and CH_2; 1383 cm^{-1}, δCH_3; 1298, 1185, 1108, 1042–1039 cm^{-1}, νC–O of aromatic ether; 915 cm^{-1} of terminal epoxy group and 832–827 cm^{-1} δCH of aromatic ring. These absorption bands due to their positions, shapes and intensities indicate that the sizing material of the 6k carbon fiber is an epoxy resin based on bisphenol-A and epichlorohydrin [9]. For the 12k carbon fiber the absorption band in 1722 cm^{-1} (νC=O) is higher than the one observed for the 6k, which indicates the presence of an epoxy resin and an ester and/or an epoxy resin modified by ester groups [10].

Carbon fiber 6k and 12k surfaces after EB irradiation were brighter, in comparison to their surfaces before irradiation, which were opaque. This fact indicates modifications on their surface characteristics. This effect was observed for both carbon fibers studied.

The visual aspect of the test specimens after breakage prepared from irradiated carbon fibers and non-irradiated carbon fibers was completely different. This is shown in Fig. 1. Test specimens

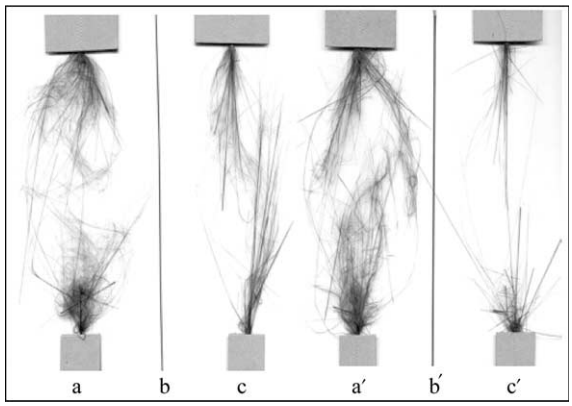

Fig. 1. 6k carbon fiber test specimens: non-irradiated, after breakage (a), before tensile test (b), and irradiated with 300 kGy, after breakage (c). 12k carbon fiber test specimens: non-irradiated, after breakage (a′), before tensile test (b′), and irradiated with 300 kGy, after breakage (c′).

from non-irradiated 6k and 12k carbon fibers, respectively named a and a′ in the figure, present a higher disarranged aspect than the test specimens prepared from irradiated 6k and 12k carbon fibers, respectively named c and c′ in the figure. Test specimens prepared from non-irradiated carbon fibers present a fiber distribution with many separated filaments giving to them a very disordered aspect. On the other hand, test specimens prepared from irradiated carbon fibers present a high number of fragments containing some bonded filaments, which gives to them a very organized aspect. These facts denote a better adhesion between the fiber and the matrix in the test specimens prepared from irradiated carbon fibers.

SEM micrographs obtained for samples after breakage confirmed these observations. The micrographs are shown in Fig. 2. For test specimens prepared from non-irradiated 6k and 12k carbon fibers, respectively named a and a′, it is observed that the fibers are separated, indicating a poor adhesion between them and the matrix. On the other hand, for test specimens prepared from irradiated 6k and 12k carbon fibers, respectively named b and b′, the micrographs show uneven surfaces and a high content of fibers bonded, indicating a better adhesion [11]. Also, the matrix amount around the carbon fibers is higher for test specimens prepared from irradiated fibers than to the

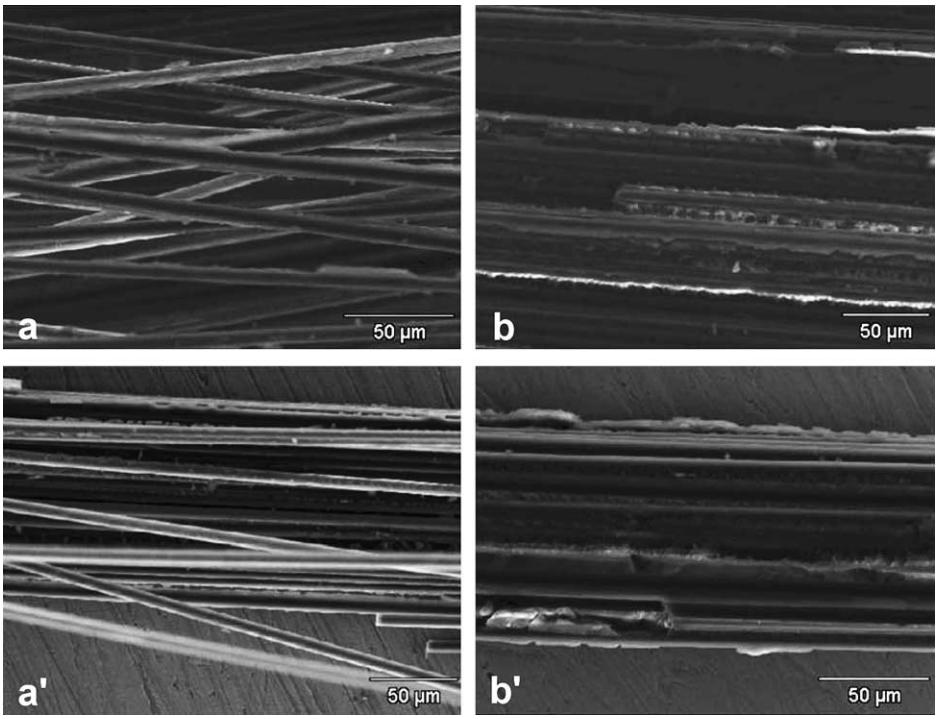

Fig. 2. SEM micrographs of 6k and 12k carbon fibers after breakage for non-irradiated (a, a′) and irradiated (b, b′) samples, respectively.

ones prepared from non-irradiated fibers. The fact that the micrographs obtained for test specimens prepared from irradiated carbon fibers had presented poor resolution is due to the content of matrix bonded to the carbon fibers in this case. Since the samples were not coated with a conducting material prior to examination, the high matrix content gives a reflexive surface.

Table 1 shows tensile properties data obtained for 6k and 12k resin-impregnated specimens. Tensile strength values for 6k and 12k specimens are different due to the number of filaments present in each kind of fiber, consequently tensile strength values obtained are higher for 12k, independently of the EB irradiation. From Table 1 data it have been also observed that tensile strength values for all test specimens prepared from irradiated carbon fibers, present a slight tendency to increase compared with the values obtained for test specimens prepared from non-irradiated carbon fibers (0 kGy). However, these values can be considered constant in the range of radiation dose employed.

Table 1
Tensile properties data for 6k and 12k carbon fibers

Dose (kGy)	6k		12k	
	TS (MPa)	*E* (GPa)	TS (MPa)	*E* (GPa)
0	(3230 ± 147)	(223 ± 6)	(4425 ± 145)	(232 ± 9)
50	(3472 ± 122)	(221 ± 8)	(4781 ± 139)	(227 ± 6)
100	(3582 ± 150)	(223 ± 8)	(4608 ± 65)	(230 ± 9)
200	(3373 ± 160)	(212 ± 5)	(4662 ± 215)	(238 ± 6)
300	(3490 ± 174)	(222 ± 7)	(4747 ± 183)	(226 ± 8)

The same behavior was observed for both 6k and 12k carbon fiber test specimens. On the other hand, these irradiation conditions had no effect on the Young's modulus for all samples tested.

All the previous observations indicate that the EB radiation promotes a fiber surface modification, which causes an improved adhesion between the fiber and the matrix used to prepare the test specimens. This behavior was observed for both carbon fibers studied, independent of the differences in the sizing materials and number of filaments.

4. Conclusion

The evaluation of the obtained results has shown that EB radiation promoted modifications on the carbon fiber surface. This was verified in the irradiated carbon fiber specimens by: the brightness of the carbon fiber surface after EB irradiation, the modifications in the test specimens aspects after breakage and the slight tendency to an improvement in the tensile strength data, compared to the behavior observed for test specimens prepared from non-irradiated carbon fibers. These modifications were similar for all applied doses, from 50 to 300 kGy, but they were not observed in test specimens prepared from non-irradiated carbon fibers. The effects were the same for 6k and 12k carbon fibers, for both sizing materials.

Based on the obtained results, it was not possible to draw a conclusion on whether the modifications promoted by EB irradiation are related to the fiber surface (finishing), the sizing composition or both.

The obtained results suggest also that the modifications induced by EB irradiation improve the adhesion between the carbon fibers and the matrix, as it was observed in the SEM micrographs.

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References

[1] M.M. Schawartz, Composite Materials Handbook, McGraw-Hill, New York, 1984, p. 1.2.
[2] K.K. Chawla, Composite Materials: Science and Engineering, Spring-Verlag, New York, 1987, p. 79.
[3] R.L. Clough, Nucl. Instr. and Meth. B 185 (2001) 8.
[4] A. Berejka, C. Eberle, Radiat. Phys. Chem. 63 (2002) 551.
[5] B. Zsigmond, L. Halász, T. Czvikovszky, Nucl. Instr. and Meth. B 208 (2003) 247.
[6] ASTM Standard, D4018-99.
[7] ASTM Standard, E83.
[8] ASTM Standard, D792.
[9] N.P.G. Roeges, A Guide of Infrared Spectra of Organic Structures, John Wiley & Sons, New York, 1981.
[10] D.O. Hummel, F. Scholl, Atlas of polymer and plastics analysis, Vol. 2aI–2aII, Verlag Chemie GmbH D-6940 Weinheim, 1984.
[11] H. Kudoh, T. Sasuga, T. Seguchi, in: R.L. Clough, S.W. Shalaby (Eds.), Irradiation of Polymers, American Chemical Society, Washington, 1996, p. 201.