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Effects of Electron Beam Irradiation on Carbon Fiber Tensile Properties

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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 resin-impregnated specimens obtained from two commercial carbon fibers (6k and 12k). EB radiation was applied in two conditions: on the carbon fiber itself before preparing resin-impregnated specimens for mechanical testing and directly on the final test specimens. Experimental results showed that EB irradiation improved the tensile strength of carbon fibers in the studied conditions for test specimens prepared from irradiated fibers. Furthermore, SEM observations have shown that EB irradiation promoted significant changes in the failure mode.

1. Introduction

Composite materials are systems composed of two or more macro constituents differing in form and/or material composition that are essentially immiscible in each other [1]. Polymeric composites are made of an organic matrix and a reinforcement material (fibers). Carbon fibers are used associated to epoxy matrix in advanced composites. These materials are frequently used for structural purposes in conditions of high mechanical loads.

An important factor to assure the good performance of carbon fiber based composite materials is the adhesion between the surface of the carbon fiber and the epoxy matrix [2]. Commercial carbon fibers present a sizing material on their surfaces in order to protect the filaments and to improve the adhesion with polymeric matrixes. Therefore, this material has an important participation 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. For the studied material, EB radiation could promotes excitation reactions on the fiber/matrix interface resulting in improved adhesion properties [5].

The aim of this paper was to evaluate the effects of EB radiation on the tensile properties of resin-impregnated specimens obtained from two different commercial carbon fibers (different number of filaments per roving) used for structural applications in two conditions: on the carbon fiber itself before preparing resin-impregnated specimens for mechanical testing and directly on the final test specimens.

2. Experimental

2.1. Samples

Two different commercial carbon fibers roving of high tensile strength were studied. One carbon fiber roving contained 6 000 elementary filaments (6k) and the other one with 12 000 filaments (12k). The sizing material on the 6k carbon surface was an epoxy resin based on bisphenol-A and epichlorohydrin and on the 12k, an epoxy resin modified by ester groups [6].

2.2. EB irradiation conditions

EB irradiation was carried out at the IPEN 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}. Overall doses applied were 50, 100, 200 and 300 kGy. EB radiation was applied in two kinds of samples: on the carbon fiber itself before preparing test specimens and directly on test specimens.

2.3. Mechanical tests

Tensile properties of the carbon fiber rovings were determined as resin-impregnated thermal cured specimens according to ASTM D4018 [7]. This procedure is used to evaluate tensile strength (TS) and Young's modulus (E) in continuo filament carbon fiber due to the difficulties to perform mechanical tests directly with filaments. In this way, the results obtained from impregnated and consolidated fiber samples can be considered the TS and E values, which are available in the material when used as intended.
The resin formulation for the impregnation was a conventional epoxy system for thermal cure at a maximum temperature of 130^{o} 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 [8]. Experimental data obtained for each type of fiber were TS and elongation at breakage. Volumetric fiber densities had been previously determined by a liquid displacement technique [9] and from these values, it was calculated the linear densities of the fibers. From the results of load and elongation and the values of linear and volumetric densities, TS and E were calculated as described in [7] for each type of fiber. A set of specimens of each carbon fiber roving (without having been EB irradiated) was used as blank. Mechanical data obtained for each condition are an average of the measurements with six specimens.

2.4. Scanning Electron Microscopy (SEM)

SEM micrographs of the fiber surfaces from fractured samples were obtained using a scanning electron microscope model JXA-6400 (JEOL).

3. Results and Discussion

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

Tables I and II show the results obtained, respectively, for test samples which were irradiated before preparing test specimens (IBP) and after preparing test specimens (IAP). The sample identified as blank in the tables corresponds to the carbon fiber roving without EB irradiation. 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 conditions.

TABLE I: TENSILE PROPERTIES DATA FOR 6k AND 12k CARBON FIBERS IRRADIATED BEFORE PREPARING TEST SPECIMENS (IBP).

Sample	6k		12k	
	TS (MPa)	E (GPa)	TS (MPa)	E (GPa)
blank	3230	223	4425	232
50 kGy IBP	3472	221	4781	227
100 kGy IBP	3582	223	4608	230
200 kGy IBP	3373	212	4662	238
300 kGy IBP	3490	222	4747	226

Table I data show that there is an improvement in the tensile strength within the radiation dose applied for IBP samples when compared to the blank values. This improvement is well beyond the range of experimental errors. However, there were no significant changes in the values obtained for IBP samples considering the range of radiation dose employed. The effect was similar for both carbon fibers studied, regardless of the differences of its sizing materials.

TABLE II: TENSILE PROPERTIES DATA FOR 6k AND 12k CARBON FIBERS IRRADIATED AFTER PREPARING TEST SPECIMENS (IAP).

Sample	6k		12k	
	TS (MPa)	E (GPa)	TS (MPa)	E (GPa)
blank	3230	223	4425	232
50 kGy IAP	3184	215	4457	229
100 kGy IAP	3285	216	4520	232
200 kGy IAP	3323	224	4385	227
300 kGy IAP	3320	222	4404	228

Table II data show that 6k and 12k IAP test specimens present values of tensile strength of the same magnitude as the one obtained for the blank. Therefore, EB irradiation do not have effects on the carbon fiber tensile properties when it is applied directly on the prepared test specimens.

Table I and Table II data show that the irradiation conditions had no effect on the Young's module (E) for all samples tested.

There were also observed changes in the aspect of test specimens after breakage. The visual aspect of test specimens after breakage prepared from irradiated carbon fibers (IBP) and non-irradiated carbon fibers (blank and IAP) was completely different. Test specimens after breakage 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.

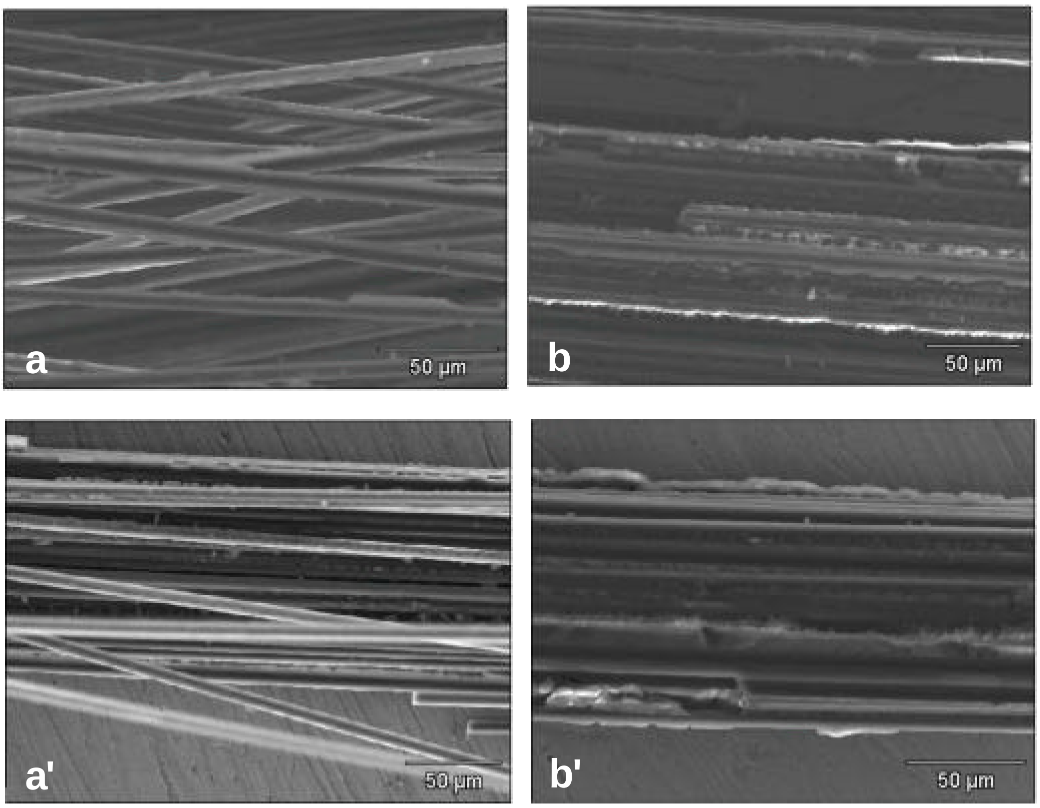

FIG. 1. SEM micrographs of 6k and 12k carbon fibers after breakage for non-irradiated (a, a') and irradiated before preparing test specimens (b, b') samples, respectively.

SEM micrographs (*Figure 1*) confirmed this fact, the micrographs obtained for IBP test specimens showed uneven interfaces, fibers bonded and higher resin content around them, what is an indication of a better adhesion, as it was observed in [10]. For the blank and IAP samples, the images obtained showed separated fibers, denoting a poor adhesion between fiber and matrix. For IAP samples irradiated with 300 kGy, the specimens after breakage, presented different characteristic when compared to the other doses applied. This means that, with this dose, it was possible to increase the degree of reticulation in the matrix. However, there was no significant improvement in the fiber tensile strength, because under this condition it was not possible to promote modifications on the fiber surface in order to obtain a better adhesion.

4. Conclusion

Tensile strength improvements and test specimens aspect after breakage for IBP samples suggest that the modifications induced by EB irradiation promote changes on the fiber surface, resulting in a better adhesion between the carbon fibers and the resin used to prepare test specimens [5]. This effect is an important factor to improve the mechanical performance of composite obtained from these materials.

The improvement obtained was very similar in the range of applied doses, from 50 kGy to 300 kGy. However, it was only observed for test specimens prepared from irradiated carbon fibers. This behavior was observed for both carbon fibers studied, independent of the differences in the sizing materials and number of filaments.

The fact that the change observed for IAP test specimens after breakage only was detected with a dose of 300 kGy means that in order to increase the degree of reticulation of the thermal cured matrix it is necessary to apply a high dose of radiation. On the other hand, to induce modifications on the carbon fiber surface the lowest dose applied (50 kGy) was enough.

5. References

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