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MODIFICATION OF POLYPROPYLENE/GLASS FIBER COMPOSITE BY ELECTRON BEAM IRRADIATION

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Abstract. *The properties, such as high chemical and corrosive resistance, ease moldability and the possibility to design complex parts, allowing the polymers to be extensively applied in many products. However, in specific applications, some properties of the polymers need to be modified to suit the material for the mandatory requirements. The ionizing radiation process, which uses electron beam equipment, represents a valuable alternative to conventional chemical methods, with the purpose to improve the characteristics and properties of the polymers. Electron beam (E-beam) irradiation can modify the molecular structure of the polymers, resulting mainly in current branching and crosslinking. These phenomena cause changes in the physical, chemical, mechanical and thermal properties of the polymers. This research aimed to analyze the influence of E-beam ionizing radiation on mechanical and thermal properties of samples of polypropylene/glass fiber composite dogbone tensile bars samples. The samples have been subjected to different doses of E-beam radiation and were evaluated for mechanical properties (tensile strength) and thermal (resistance to an incandescent wire) properties. The results were compared to non-irradiated samples. The tensile strength was not modified by the different doses of ionizing radiation applied to the samples. The resistance to the glow wire had a good performance in irradiated samples.*

Keywords: *polypropylene, electron beam, ionizing radiation, mechanical, thermal properties.*

1. INTRODUCTION

Polymeric materials are widely used for various engineering purposes. Polypropylene (PP) is one of the most versatile thermoplastics available for applications, with or without additions of charges. This polymer is a material with good properties such as low density (0.95 g / cm^3), high melting temperature (160øC), low electrical conductivity, chemical inertia, low water absorption (0.02%) and Low cost (Maddah, 2016).

PP is the second most traded polymer in the world after polyethylene (PE). The global demand for polymers is about 218 million tons wherein the polypropylene share consumption is about 25%. Among the various types of PP, the homopolymer is commonly used in injection mold grades for several applications, being parts of components for automobiles, appliances, electrical and others (Plastemart, 2014). This broadly ranges of applications makes the PP an important polymer for researches and developments in the industry and universities, including those glass fiber reinforced grades.

The effects of the ionizing irradiation prior on the molecular structure of the polymers, such as crosslinking and degradation are widely studied (Charlesby, 1985; Goel, et al., 2013). Ionizing radiation (gamma rays or E-beam) has been used as a very effective means of improving some characteristics of the final parts. The E-beam irradiation process offers several advantages when compared to other types of radiation, e.g., γ-rays and x-rays. The E-beam is faster and more accurately controlled. Radioactivity is not permanent in the E-beam process, since the principle of the machine works by electron acceleration, what differs from gamma rays, which emitted constant radiation by a radioactive element (BGS, 2017).

The E-beam irradiation process is practically free of residues, conferring a great appeal to sustainability. The radiation absorbed by the part treated may confer, large changes in the properties of stable polymers, due to crosslinking and degradation of molecular chains. Irradiation doses can adjust the level of crosslinking. An important difference between E-beam and gamma rays is that E-beam has a lower radiation penetration in the thickness of parts treated, which needs to be carefully calculated to assure the quality of the process. A mean dosage can be applied in seconds using electron accelerators, whereas several hours are required in the gamma radiation plant (Drobny, 2003; BGS, 2017). Electron beam irradiation interacts with the polymers transferring energy to the atoms of the polymer chain and causing changes in its structure, altering its physicochemical properties. Studies show that these modifications result in crosslinking and degradation involving the radiation process (Kabanov et al., 2009).

The uses of ionizing radiation in polymers are gaining the industry attention, for economic reasons, production costs and product lifetime. These improvements of the properties depend on the polymer used and the dosage of irradiation, since each material has a specific behavior. It is possible to irradiate food packaging, medical appliances, automotive products and a wide range of other applications (Zagorski, 2013). Several studies showed that no additives are needed for ionizing polypropylene to have an effective radiation (Krause, et al., 2006).

The purpose of this study is to analyze the modification in mechanical and thermal properties of a glass fiber reinforced PP based composite treated by electron beams ionizing radiation.

2. EXPERIMENTAL PROCEDURE

A glass fiber reinforced polypropylene based composite was used for this study due to the good mechanical properties and low costs when compared to other polymers. The material used this study is a PP resin, 30% glass fiber reinforcement, halogenated flame retardant and hot stabilizer for injection molding.

Samples were produced according to ASTM-D638, type I (ASTM, 2014) by injection molding an Arburg 420C 1000-250 machine and were submitted to E-beam process. The irradiation was performed in50, 100, 150 and 200 kGy doses. The E-beam machine used for this process has a capacity up to 10 MeV, 60 kV and 5 mA. A comparison was made between samples with and without irradiation in different doses, evaluating the mechanical and thermal properties.

The mechanical evaluation was performed in a universal MTS test machine, model E45.305, by tensile tests. An extensive video was used for measuring the specimens' strain, a 300 kN load cell and 1 mm/m speed (several speeds were tested, and the best fitted was 1mm/m). For each condition, ten specimens were evaluated. Young's modulus, yield, tensile strength and strain values were determined.

The thermal properties were determined by a test, called resistance to an incandescent wire, which was developed specifically to verify the irradiated samples. The test consists in heating an incandescent wire tip to a temperature of 180± 5 °C and applies to the samples for 10 seconds. Fig. 1 illustrates the schematic representation of the device and method for checking irradiated samples.

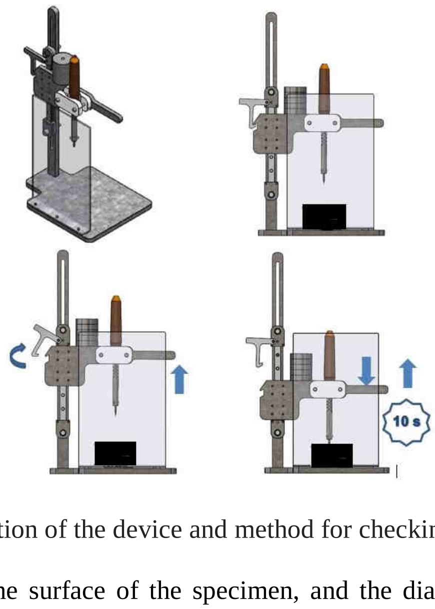

Figure 1. Illustration of the device and method for checking irradiated samples.

The tip leaves an impression on the surface of the specimen, and the diameter of the impression was measured through of a three-dimensional coordinate measuring machine (MMC Crysta Plus M 574, Mitutoyo) and tip penetration assessed through a 3D multi-sensor (Optiv Performance, with PC-DMIS Vision Measurement Software, HEXAGON

Manufacturing Intelligence). The sample was considered approved when the impression on its surface has a diameter of less than 3 mm and does not pierce its thickness. The sample fails the test if the impression on its surface has more than 3 mm or perforates its thickness.

3. RESULTS AND DISCUSSION

The samples evaluated were irradiated at different doses up to 200 kGy. By comparing the visual aspect, a slightly yellowish region could be observed, which intensifies with increasing radiation doses, how can be visualized in the Fig. 2. This yellowish is a result of a series of modifications in the polymer, like chains scissoring. According to Clough et al. (1996), the ionizing radiation can cause changes a color in polymers, conferring two different types of color centers. Have two reasons for this: the permanent ones, which correspond to a static aeration of the molecular structure of the material, and the unstable ones, which are associated with free radicals trapped in the polymer structure and that may disappear in some conditions.

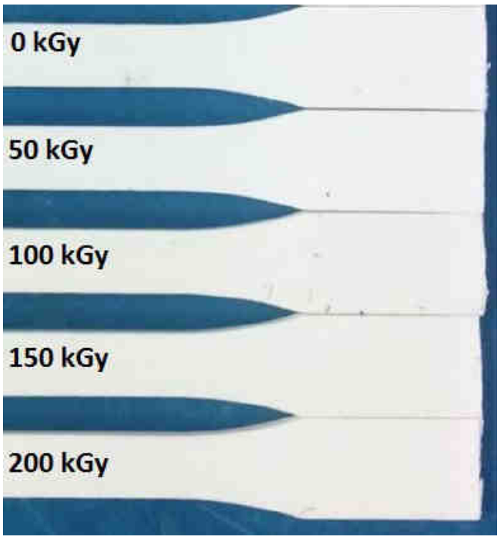

Figure 2. A slightly yellowish region could be observed samples.

Tab. 1 shows the values found for tensile tests. The results of the tests showed a small decrease in tensile strength (UTS) at the dose of 200 kGy, around 4.83% and inversely an increase in its Young Modulus (E) which was 2.08%.

The greatest difference found was that elongation (strain) varied from 3.01% for a sample without irradiation and 1.55% for a 150 kGy sample, what represents a reduction of 48.50%. A significant reduction, when compared with the 2% value defined in the data sheet, showed by the supplier. The material without irradiation presented a higher value of elongation.

Table 1. Mechanical Properties of non-radiated and radiated polypropylene/glass fiber composite at different doses.

Dose (kGy)	E [MPa]	UTS[MPa]	Yield [MPa]	Strain [%]
0	7586	62	47	3.01
50	7791	62	48	2.49
100	7831	62	48	2.38
150	7813	61	48	1.55
200	7817	59	48	1.72

Fig. 3 shows a comparison of the tensile strength results (stress *vs.* strain curves) from non-irradiated up to 200 kGy irradiated samples of polypropylene/glass fiber composite (PP30% GF). It is observed in the figure that the elongation decreases when the irradiation doses increase.

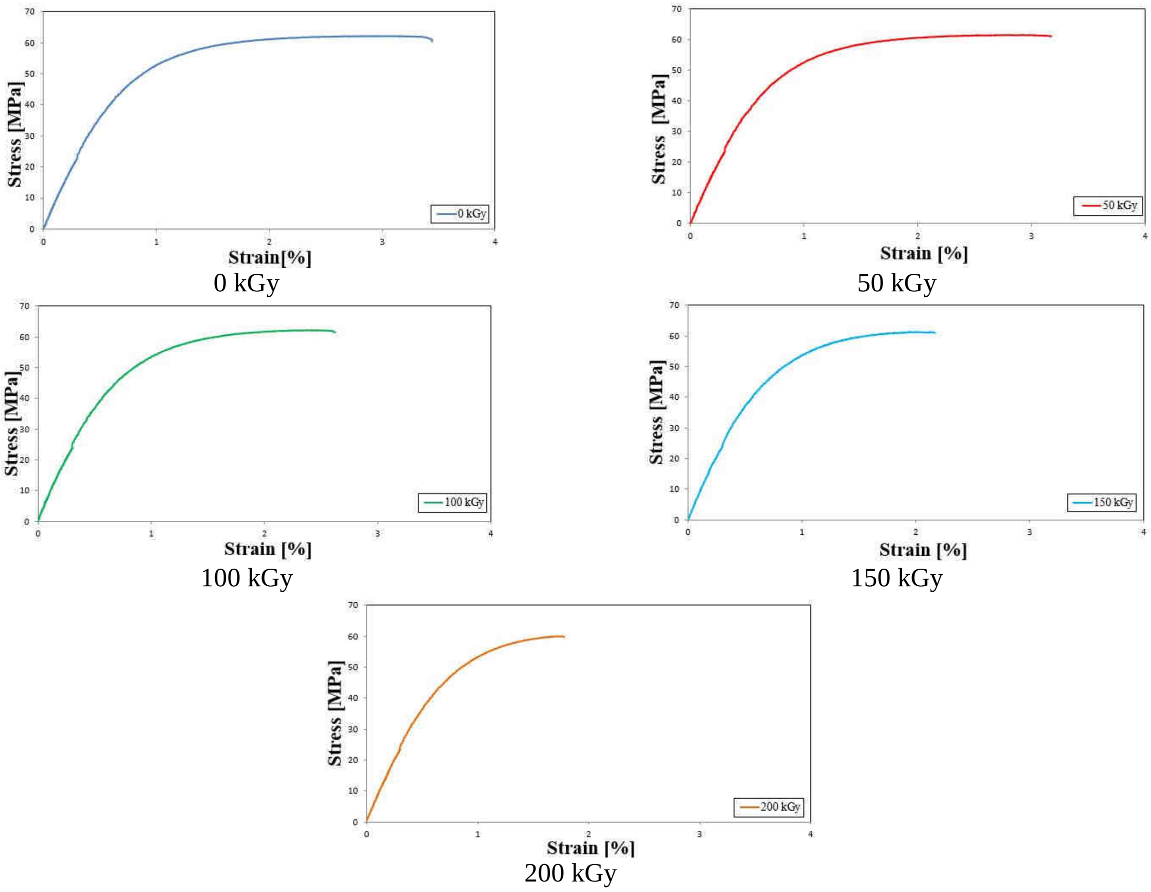

Figure 3. Comparison between tensile tests results Stress vs. Strain for each sample.

Fig. 4 is a graphic displaced on the y-axis for a better visualization of the difference in Stress vs. Strain curves. There is clearly seen a reduction in elongation as the irradiation dose increases. Comparing the values obtained in the tests carried out in this paper and a polypropylene without fiber and without flame retardancy studied by Souza, et al. (2009), it can be evidenced that there were no significant changes and that the additives and reinforcements contribute to material to maintain its mechanical properties, however with a decrease in the elongation.

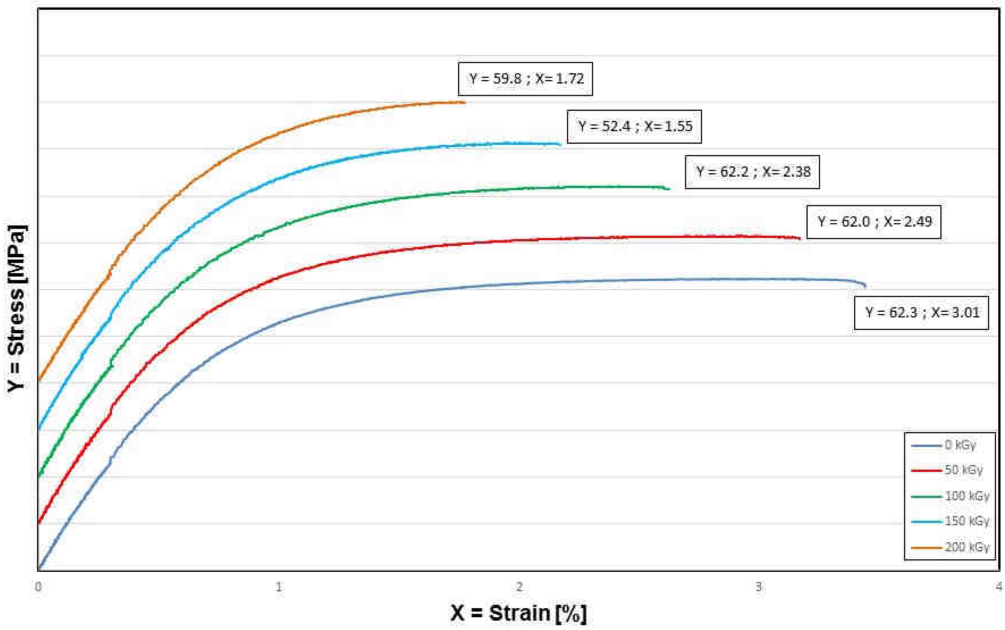

Figure 4. Comparison between tensile tests results (Stress vs. Strain) for different irradiation doses.

In the results in the resistance to the incandescent wire test, the irradiated samples presented a better performance at 180 °C comparing to the non-irradiated sample (0 kGy) within doses of 50 and 100 kGy. In the non-irradiated sample (0 kGy) was observed that there a greater displacement of the material occurs around the tip, upward above the surface of the sample, during the thermal test performed. At the end of the test, this fact is assimilated to the larger diameter of the impression in the sample. Regarding penetration, the sample without irradiation presented a lower depth. This phenomenon occurs due to the non-irradiated material has a greater flowability and ease movement of the chains during it is melting, due to no crosslinking between its molecules are presented. The values found for the penetration depth of the non-irradiated and irradiated samples are shown in Tab. 2.

Table 2. Depth of penetration for the different irradiation doses.

Dose (kGy)	Penetration [mm]
0	1.480
50	1.578
100	1.667
150	1.675
200	2.206

As the radiation increases to 50 kGy and 100 kGy, a greater crosslinking and some degradation occur, improving the temperature resistance of this material, reflected in the result. At doses of 150 and 200 kGy it is noticed that the penetration of the tip were deeper the values found in this study. This effect is due to degradation occurred in the material by the excess exposure of the material to an ionizing radiation, (Buttafava et al., 2002). In the present paper, it is possible to determine the optimized dose of radiation for best crosslinking and the degree of degradation of this polymeric material. Furthermore, degradation results in a reduction of the molecular mass and decrease of the physical properties of the polymer (Charlesvy, 1985). Within the established rules for the test, all the irradiation samples were approved, as observed in Fig.5. The penetrations in the samples were compared through a 3D multi-sensor (Optiv Performance, with PC-DMIS Vision Measurement Software). In Tab. 3 are the values found for the penetration diameter of the samples without irradiation and submitted to radiation.

Table 3. Penetration diameter for different irradiation doses.

Dose (kGy)	Penetration [mm]
0	1.25
50	1.20
100	1.20
150	1.47
200	1.55

In Fig. 5, it is possible to view that the penetration diameter in the sample without irradiation is higher than in samples irradiated with 50 and 100 kGy. It is noted that the diameter of the penetration increased as the irradiation dose increased. The degradation may cause this increase occurred during the irradiation process in which it interacted with the polymer matrix and the glass fiber, in addition to the interaction with additives, such as flame retardants which are acting on the polypropylene/glass fiber compound.

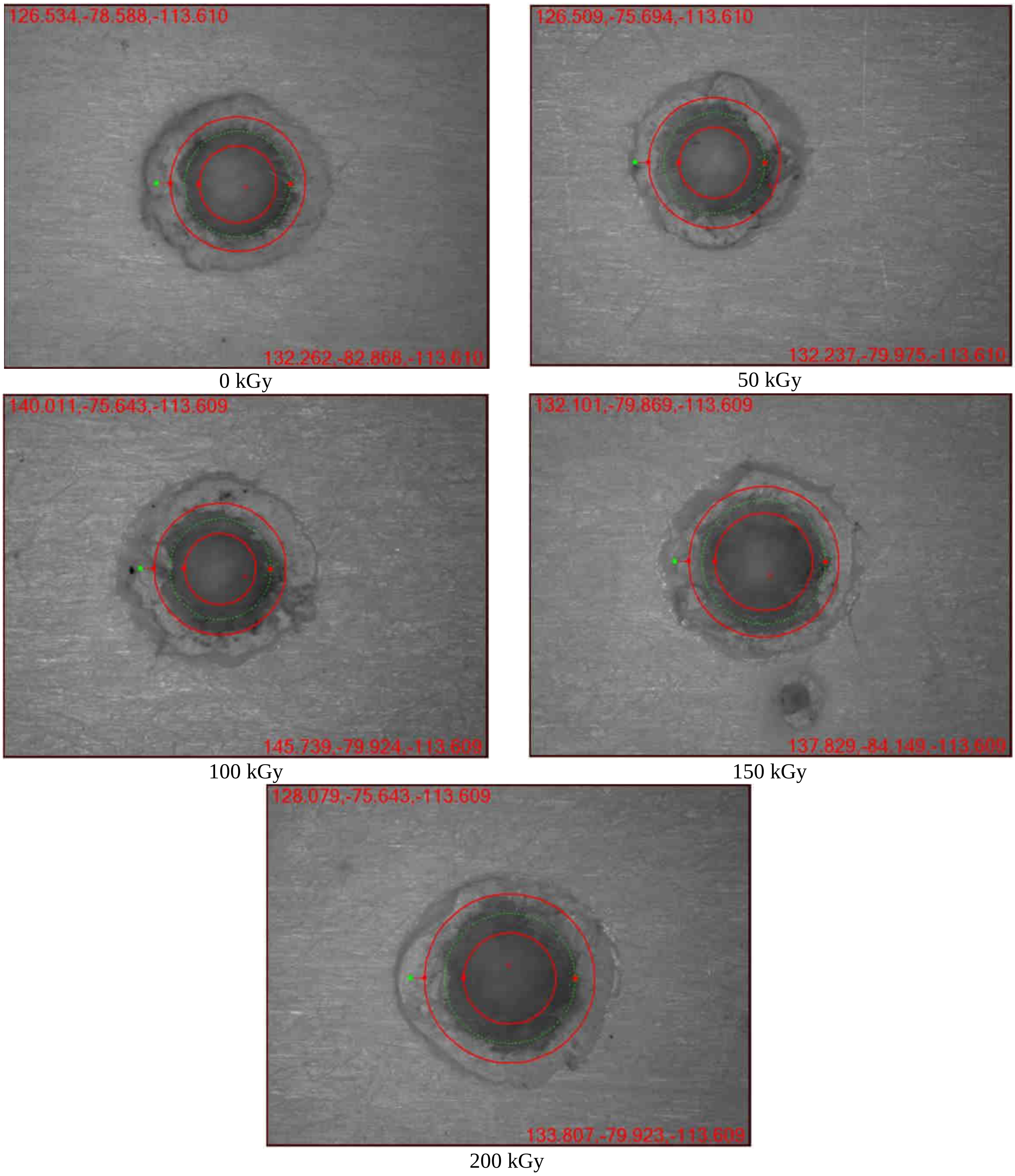

Figure 5. Comparing Glow Wire different irradiation doses.

4. CONCLUSIONS

The samples were subjected to different doses of E-beam irradiation, and the mechanical and thermal properties were compared to the non-radiated samples.

The mechanical properties of the polypropylene/glass fiber composite are little affected by exposure to electron beam radiation. The greatest difference found was the elongation for the 150 kGy sample, which presented a reduction of 48.50%. A significant reduction is comparing with the theoretical value offered by the supplier which is about 2%.

In the thermal tests, the samples had a good performance in the resistance to the incandescent wire test, suggesting that the radiation. In addition to crosslinking of the polymer chains of the composite, could somehow have interacted with the glass fiber and possibly with the flame retardants. The best results were found for 100 kGy samples.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

The authors M.A. Castro, R. Michalak, D.C. Pfeifer and D.O.S Recouvreux are the only responsible for the printed material included in this paper.