Effect of Ionizing Beta Radiation on the Strength of Bonded Joints and Adhesive Properties

M. Bednarik, D. Manas, M. Manas, M. Stanek, A. Mizera, M. Ovsik, and P. Kratky

***Abstract*—** In this study there was found that ionizing beta radiation increased the strength of bonded joints and improved the adhesion properties of linear low-density polypropylene (LLDPE) and polyamide 6.6 (PA 6.6). Generally, for the formation of quality bonded joint it is important to wet the adhesive bonding surface well. Wettability is characterized by the contact angle of wetting. The liquid has to have a lower surface tension than the solid in order to be able to wet the solid substance. The measurement results indicated that ionizing beta radiation was a very effective tool for the improvement of adhesive properties and increased the strength of bonded joints of studied polymers. Bonded surfaces with ionizing beta radiation doses of 0, 33, 66, and 99 kGy were irradiated. The best results were achieved by irradiation at doses of 66 and 99 kGy by which the highest surface energy and the highest strength of bonded joints of LLDPE and PA 6.6 were achieved.

***Keywords*—**Surface energy, ionizing beta radiation, strength, bonding, adhesion, polymers.

I. Introduction

BONDING has experienced tremendous expansion in the field of joining materials in the last years. Because of that expansion bonding is classed as new techniques even when it is in fact very old.

In comparison with conventional joining methods (riveting, welding and screwing) bonding provides a new combination of options and it allows obtaining special shapes and properties which cannot be created by conventional methods of coupling. Joining materials using adhesive joints offers several benefits, but also limiting factors if compared with using mechanical joints. To decide about the type of coupling it is necessary to consider the advantages and disadvantages of bonding in comparison with traditional joining technique. [2, 4, 5, 9]

The advantage of bonded joints is that we can connect the same and dissimilar materials and do not take into account the thickness. Application of adhesives does not undermine the coherence of the connect parts and watertight and gastight joints may be prepared. It does not disturb profile or aesthetic appearance of bonded file and does not increase its weight. Joints can be transparent or colored and it is possible to achieve their high strength. [55-60]

Adhesive bonding has also several technological disadvantages, in comparison with mechanical joints. The disadvantages of bonded joints are the requirements for planeness and cleanness of surface to be bonded and sensitivity to peel stress. Special surface treatments are required for bonded materials with poor adhesion properties and maximum bond strength is reached after the certain time. [2, 12, 14]

II. Experimental

The aim of the experiment was to examine the effect of beta irradiation on the strength of bonded joints for selected types of materials.

A. Materials

For this experiment polyamide 6.6 FRIANYL A63 VN (PA 6.6) and linear low-density polypropylene (LLDPE) were used.

Polyamides are linear polymers which contain amide groups in the chain. Polyamides have very good mechanical properties and especially PA 6 and PA 6.6 are used to produce fibers. Further processing is by injection and extrusion. Extrusion from the polyamides used to produce sheets, rods, tubes and other profiles. Injection molded components in the automotive industry as suction manifold of the engine or ventilators are used.

Linear low-density polyethylene is the youngest of all types of polyethylene. Its structure is similar to the structure of HDPE but has a low crystallinity given to a large number of short chains. Linear low-density has penetrated almost all traditional markets for polyethylene. It is used to produce

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plastic bags and sheets, plastic wrap, stretch wrap, pouches and toys. [15, 16, 19]

For bonding LLDPE it is necessary to modify surfaces which will be bonded. Surface modification improves its wettability in aqueous media and also improves adhesive properties. [10, 13]

The samples were made using the injection molding technology on the injection molding machine Arburg Allrounder 420C. Injection conditions are shown in Table 1.

Table 1 Injection conditions

	PA 6.6	**LDPE**
Injection rate	40 mm/s	50 mm/s
Injection pressure	900 bar	600 bar
Injection time	0,5 s	0,4 s
Cooling time	17 s	30 s
Mold temperature	70 °C	40 °C
Holding pressure	800 bar	500 bar
Cycle time	55,5 s	56,6 s
Temperature of zone 1	220 °C	190 °C
Temperature of zone 2	250 °C	200 °C
Temperature of zone 3	270 °C	210 °C
Temperature of zone 4	280 °C	215 °C
Temperature of zone 5	310 °C	220 °C

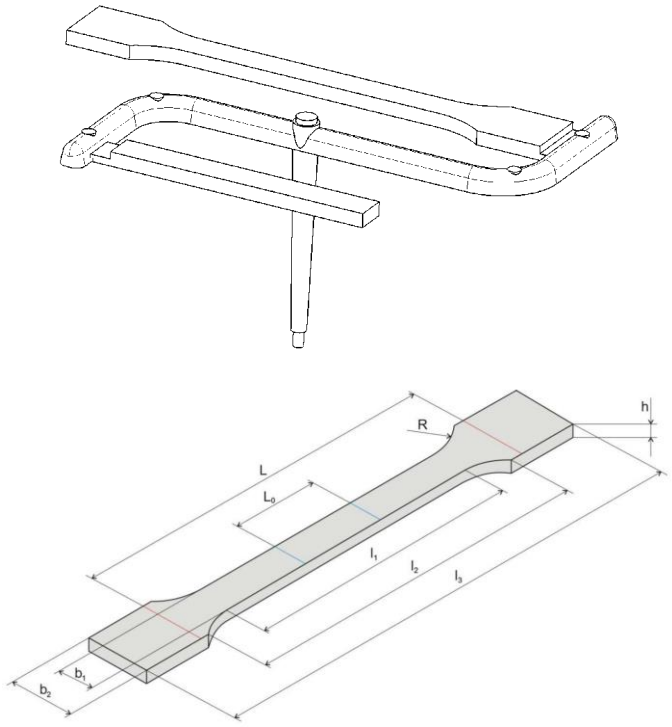

Fig. 1 Testing specimen [8]

Tab. 2 Specimen dimensions [8]

Test specimen parameters	
b_1 - Width of Gage Length	10 ± 0.2 [mm]
b_2 - Width of Gripping End	20 ± 0.2 [mm]
l_1 - Length of gage Length	80 ± 2 [mm]
l_2 - Distance Between Gripping Ends	104-113 [mm]
l_3 - Specimen Length	≥ 150 [mm]
L_0 - Distance of Extensometers	30 ± 0.5 [mm]
L - Distance of Grips	115 ± 1 [mm]
h - Specimen Thickness	4 ± 0.2 [mm]
R - Radius	20 - 25 [mm]

The samples had the shape and dimensions according to the CSN EN ISO 527 – 2 (Fig. 1 and Tab. 2). Before bonding surfaces of samples ionization beta radiation were irradiated with doses of 0, 33, 66, and 99 kGy at Beta – Gamma Service GmbH & Co. KG, Germany. [3, 6, 17]

B. Surface treatment by beta radiation

Ionizing beta radiation gives inexpensive commodity plastics and technical plastics the mechanical, thermal, and chemical properties of high-performance plastics. This upgrading of the plastics enables them to be used in conditions which they would not be able to with stand otherwise. The energy-rich beta rays trigger chemical reactions in the plastics which results in networking of molecules (comparable to the vulcanization of rubbers which has been in industrial use for so long). [1, 3, 7]

The energy from the rays is absorbed by the material and cleavage of chemical bonds takes place. This releases free radicals which in next phase from desired molecular bonds. [3, 8, 21]

The irradiation cross-linking of thermoplastic materials via electron beam or cobalt 60 (gamma rays) is performed separately, after processing. Generally, ionizing radiation includes accelerated electrons, gamma rays and X-rays. Radiation processing with an electron beam offers several distinct advantages when compared with other radiation sources, particularly γ-rays and x-rays.

The process is very fast, clean and can be controlled with much precision. There is no permanent radioactivity since the machine can be switched off. In contrast to γ-rays and x-rays, the electron beam can be steered relatively easily, thus allowing irradiation of a variety of physical shapes.

The electron beam radiation process is practically free of waste products and therefore is no serious environmental hazard. These are not only capable of converting monomeric and oligomeric liquids, but also can produce, due to cross-linking, major changes in the properties of solid polymers. The cross-linking level can be adjusted by the irradiation dosage. The absorbed dosage means the value of energy of ionizing radiation absorbed by a unit of mass of the processed material. The unit of absorbed dose is 1 Gray (1 Gy = 1J/kg). [37-46]

The main difference between beta and gamma rays is in their different abilities to penetrate the irradiated material. Gamma rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons. Due to electron accelerator, the required dosage can be applied within seconds, whereas several hours are required in the gamma radiation plant (Fig. 2). [3, 34, 35, 36]

Beta and gamma rays can be used for the irradiation of polyolefines, polyesters, halogen polymers and polyamides from the thermoplastics group, elastomers and thermoplastic elastomers. Some of them need the addition of a cross-linking agent. Polymers can be classified into two groups according to their response to ionizing radiation. One group exhibits predominant cross-linking, the other predominant chain scission. [1, 3, 34, 35, 36]

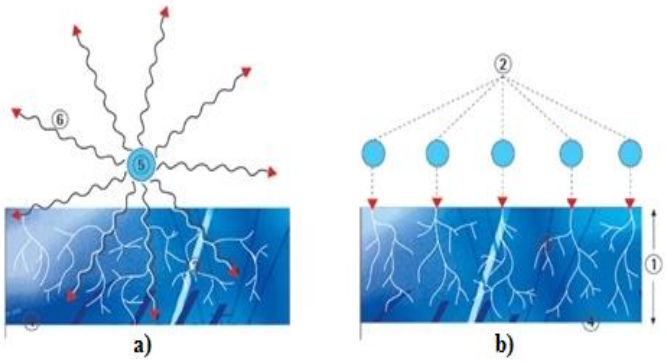

Fig. 2 Design of Gamma Rays (a) and Electron Rays (b) [3]

a) 3 – Secondary electrons, 4 – Irradiated material,
5 – Encapsulated Co – 60 Radiation source, 6 – Gamma rays
b) 1 – Penetration depth of an electron, 2 – Primary electron,
3 –Secondary electron, 4 – Irradiated material

Irradiation cross-linking of thermoplastic materials deals with creation of a cross-link among the macromolecular strings. Intermolecular forces are replaced by a covalent bond. As a result, we can optimise properties of standard and engineering polymers and impart them the properties of high performance polymers (Fig. 3). [42-54]

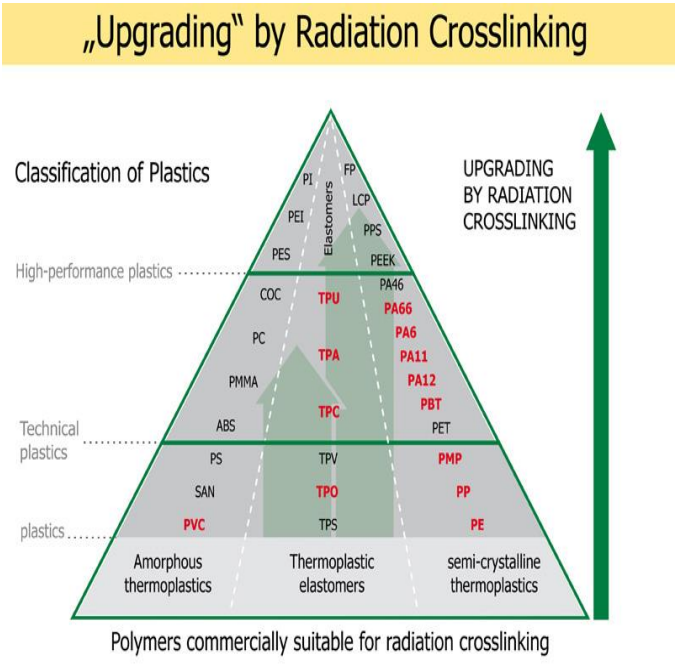

Fig. 3 Pyramid of Polymers [3]

Irradiation of polymers turned out to be interesting because of economic reasons, production costs and a life time of products. However, these benefits depend on the type of irradiated polymer and the radiation dosage. Behaviour of each material is different after irradiation. We cannot expect the

improvement in all areas (in mechanical, thermal and chemical). Most of polymers are not suitable for irradiation because of degradation and deterioration of their properties. [8, 22-33]

C. Wetting contact angle measurements

The angle of contact was measured by using the sessile drop (Fig. 4) and surface energy was determined by OWRK method (Owens – Wendt – Rabel – Kaeble method).

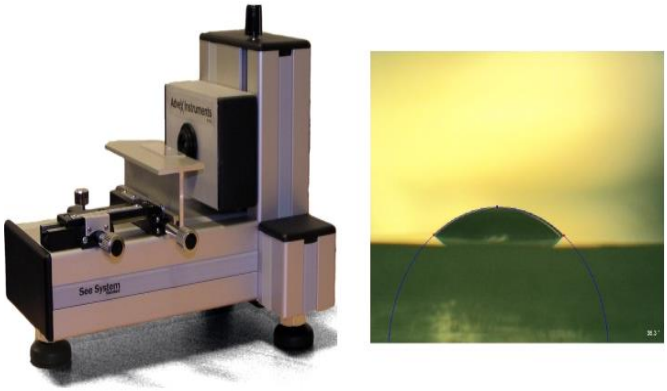

Fig. 4 Surface Energy Evaluation System (See System) [47]

The liquids water, glycerol and ethylene glycol with known γ^p (polar component) and γ^d (dispersive component) were used for calculating the surface energy of LLDPE and PA 6.6. [2, 11]

Table 3 Surface energy of liquids

Liquid	γ_l (mJ/m^2)	γ_l^d (mJ/m^2)	γ_l^p (mJ/m^2)
Distilled water	72.8	21.8	51.0
Glycerol	64.0	34.0	30.0
Ethylene glycol	48.0	29.0	19.0

The height (h) and radius (r) of the liquids were measured by using microscope and the contact angle was calculated using the following equation:

$$\text{contact angle}\,(\theta) = \frac{\sin^{-1}(2rh)}{(r^2 + h^2)}. \quad (1)$$

The polar and dispersive components of the surface energy of the polymer surface were calculated using methods OWRK:

$$\frac{(1+\cos\theta)\gamma_1}{2\sqrt{\gamma_1^d}} = \sqrt{\gamma_s^p}\sqrt{\frac{\gamma_1^p}{\gamma_1^d}} + \sqrt{\gamma_s^d}, \quad (2)$$

where θ is the contact angle of testing liquids, γ_l is the liquid surface energy, and γ_l^p and γ_l^d are the polar and dispersive components of the test liquids. The values of polar and dispersive components of testing liquids are given in Table 1. [2, 11]

Similarly, the solid – surface energy (γ_s) is expressed in terms of its polar and dispersive components:

$$\gamma_s = \gamma_s^p + \gamma_s^d. \quad (3)$$

D. Testing the strength of bonded joints

After sample preparation (production and irradiation) contact angles were measured and surface energy was calculated. Then the samples were bonded and their strength was measured. For testing the strength of bonded joints there was used a tensile test on the test machine Zwick 1456. Test conditions were according to the CSN EN ISO 527-1 and CSN EN ISO 527-2. Speed was 10 mm/min and evaluation software was Test Expert Standard. [18, 20, 34, 35, 36]

III. Results and Discussion

A. Contact angle and surface energy

The variation in contact angle of LLDPE for different doses of radiation and for different test liquids is shown in Fig. 5.

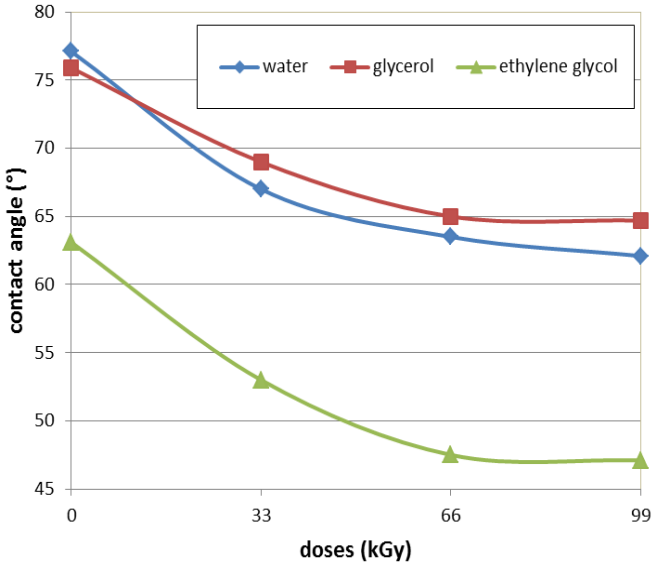

Fig. 5 Variation of contact angle with respect to radiation dose for material LLDPE

It shows that the contact angle on the untreated surface is 77.1°, 75.9°, and 63.1° for distilled water, glycerol, and ethylene glycol, respectively. The contact angle values were considerably reduced after irradiation by a dose of 99 kGy to lower values of 62.1°, 64.7°, and 47.1° for distilled water, glycerol, and ethylene glycol, respectively (referring to: Fig. 5).

The variation in contact angle of PA 6.6 for different doses of radiation and for different test liquids is shown in Fig. 6. It shows that the contact angle on the untreated surface is 61.7°, 61.6°, and 54.8° for distilled water, glycerol, and ethylene glycol, respectively. The contact angle values were considerably reduced after irradiation by a dose of 99 kGy to lower values of 56.7°, 61.3°, and 47.6° for distilled water, glycerol, and ethylene glycol, respectively (referring to Fig. 6).

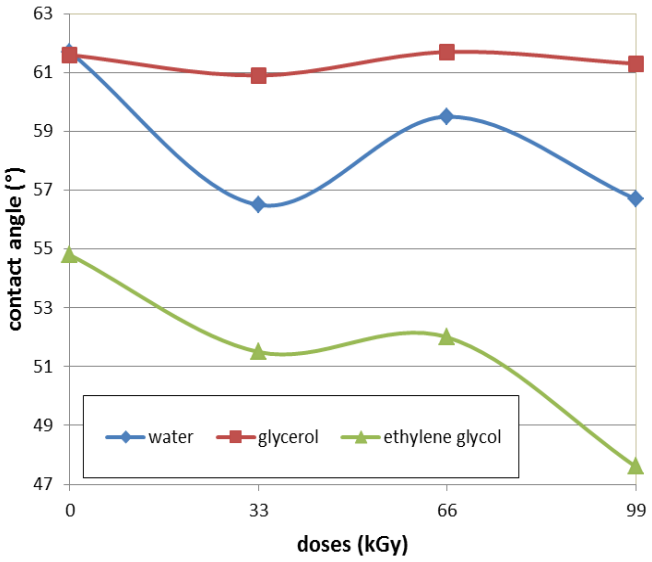

Fig. 6 Variation of contact angle with respect to radiation dose for material PA 6.6

Fig. 7 shows a plot of surface energy γ_s from the measured contact angles on the surface of the LLDPE. It shows that surface energy of untreated surface is 27.4 mJ/m^2 and 27.3 mJ/m^2 for water + glycerol and water + ethylene glycol, respectively. Ionization beta radiation increases the surface energy. The surface energy values considerably increased after irradiation by a dose of 99 kGy to higher values of 41.9mJ/m^2 for water + glycerol and 39.5 mJ/m^2 for water + ethylene glycol.

Fig. 8 shows a plot of surface energy γ_s from the measured contact angles on the surface of the PA 6.6. It shows that surface energy of untreated surface is 40.4 mJ/m^2 and 42.5 mJ/m^2 for water + glycerol and water + ethylene glycol, respectively. Ionization beta radiation increases the surface energy. The surface energy values considerably increased after irradiation by a dose of 99 kGy to higher values of 48.2 mJ/m^2 for water + glycerol and 46.2 mJ/m^2 for water + ethylene glycol.

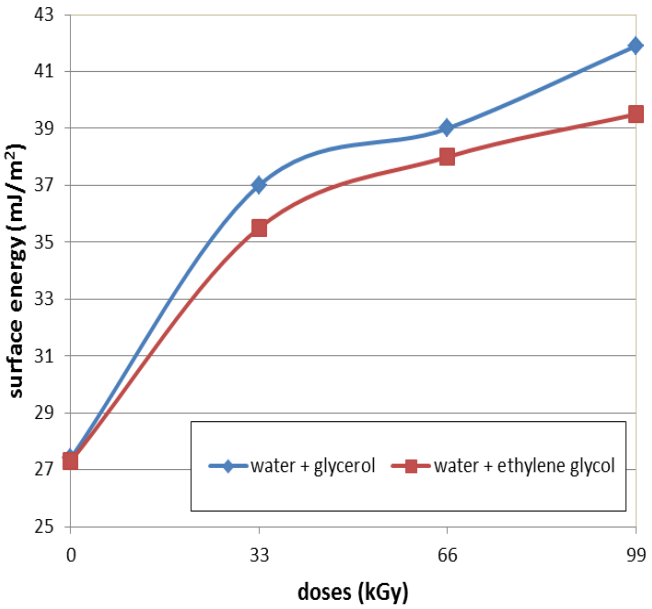

Fig. 7 Variation of surface energy with respect to radiation dose for material LLDPE

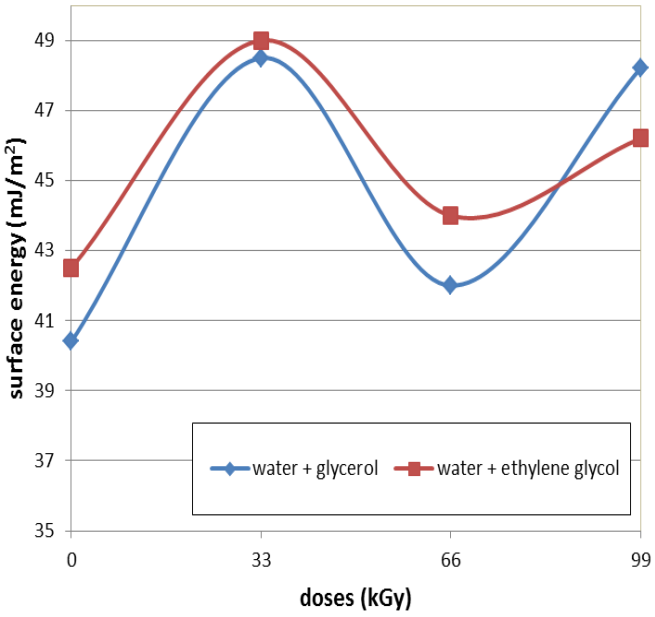

Fig. 8 Variation of surface energy with respect to radiation dose for material PA 6.6

B. Strength of bonded joints of LLDPE

Strength of bonded joints is characterized by the maximum burdensome force which endured bonded sample. For bonding of LLDPE one type of two-component methacrylate adhesive of company Plexus (Plexus MA300) and two types of cyanoacrylate adhesives of companies Cyberbond (Cyberbond 1008 and Cyberbond 2008) were used.

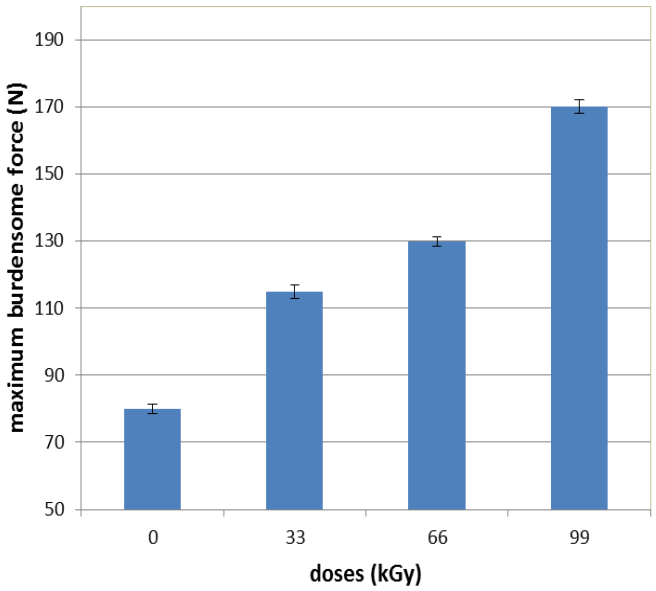

Fig. 9 Variation of strength of bonded joints with respect to radiation dose (Plexus MA300)

The highest strength of bonded joints samples of LLDPE (adhesives Plexus MA300) have those which were irradiated by a dose of 99 kGy. After the irradiation by a dose of 99 kGy strength is increased by 100 % (referring to: Fig. 9).

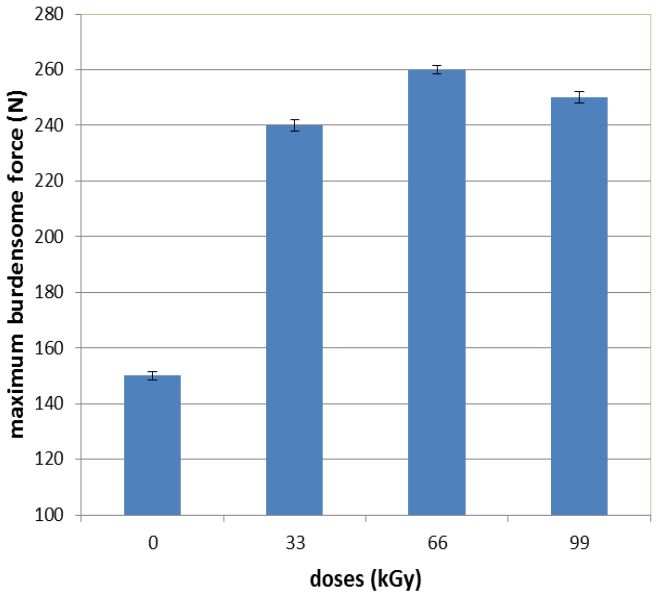

Fig. 10 Variation of strength of bonded joints with respect to radiation dose (Cyberbond 1008)

The highest strength of bonded joints samples of adhesive Cyberbond 1008 have those which were irradiated by a dose of 66 kGy. After the irradiation by a dose of 66 kGy strength is increased by 70 % (referring to: Fig. 10).

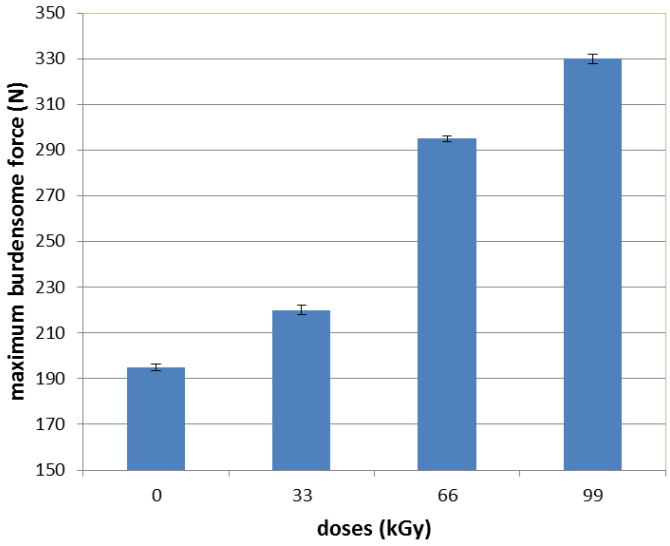

Fig. 11 Variation of strength of bonded joints with respect to radiation dose (Cyberbond 2008)

The highest strength of bonded joints samples of LLDPE (adhesives Cyberbond 2008) have those which were irradiated by a dose of 99 kGy. After the irradiation by a dose of 99 kGy strength is increased by 70 % (referring to: Fig. 11).

C. Strength of bonded joints of PA 6.6

Strength of bonded joints is characterized by the maximum burdensome force which endured bonded sample. For bonding of PA 6.6 one type of two-component methacrylate adhesive of company Plexus (Plexus MA300) and two types of cyanoacrylate adhesives of companies Cyberbond (Cyberbond 1008 and Cyberbond 2008) were used.

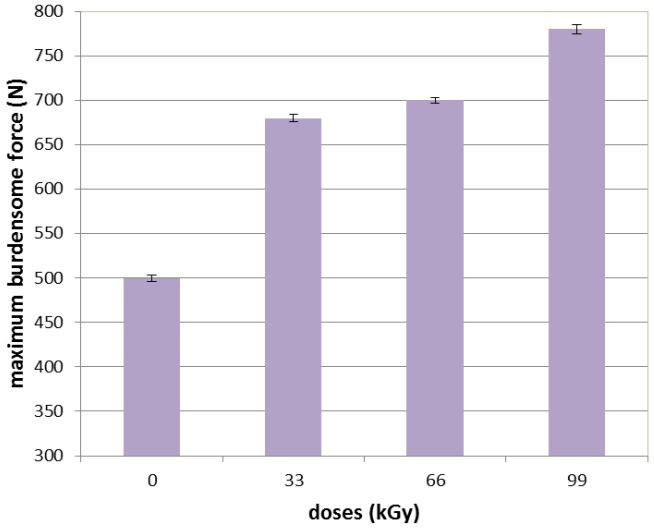

Fig. 12 Variation of strength of bonded joints with respect to radiation dose (Plexus MA300)

The highest strength of bonded joints samples of PA 6.6 (adhesives Plexus MA300) have those which were irradiated by a dose of 99 kGy. After the irradiation by a dose of 99 kGy strength is increased by 60 % (referring to: Fig. 12).

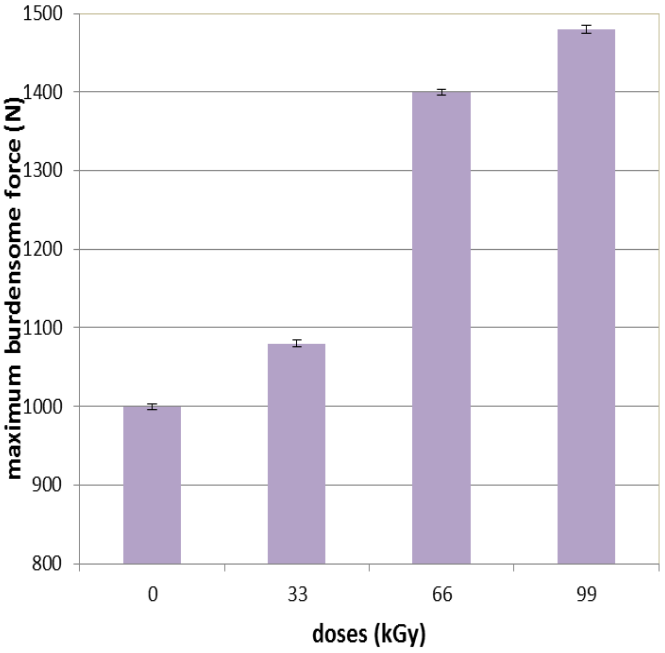

Fig. 13 Variation of strength of bonded joints with respect to radiation dose (Cyberbond 1008)

The highest strength of bonded joints samples of adhesive Cyberbond 1008 have those which were irradiated by a dose of 99 kGy. After the irradiation by a dose of 99 kGy strength is increased by 50 % (referring to: Fig. 13).

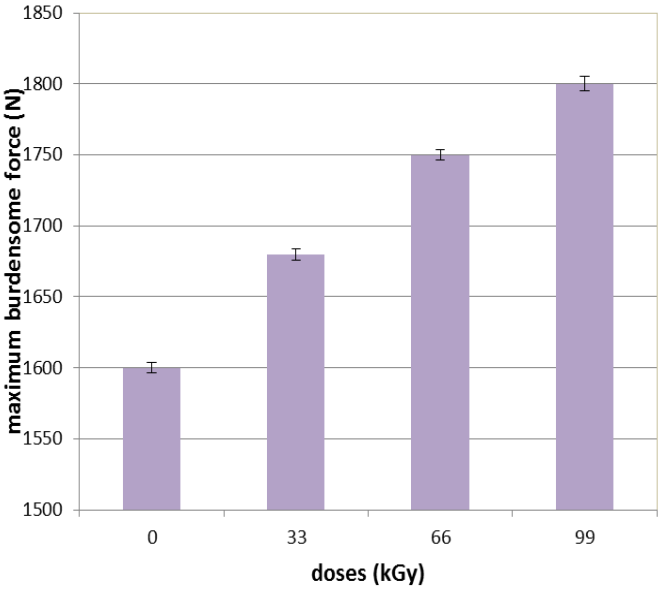

Fig. 14 Variation of strength of bonded joints with respect to radiation dose (Cyberbond 2008)

The highest strength of bonded joints samples of PA 6.6 (adhesives Cyberbond 2008) have those which were irradiated by a dose of 99 kGy. After the irradiation by a dose of 99 kGy strength is increased by 10 % (referring to: Fig. 14).

IV. Conclusion

This article describes the effect of beta radiation on the contact angle of wetting, on the surface energy and on the final strength of bonded joints of LLDPE and PA 6.6. Beta radiation increases the strength of bonded joints of LLDPE and PA 6.6 and improves their adhesion properties. The best results were achieved by irradiation at doses of 66 and 99 kGy by which the highest surface energy and the highest strength of bonded joints of LLDPE and PA 6.6 were achieved.

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