Properties of LDPE with the Filler of Recycled Irradiated HDPE by accelerated electrons

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Abstract—Irradiation of thermoplastics is a well-known modification used for improving of cheap commodity plastics; however, little research was carried-out on recyclation of such modified materials. This research paper gives possible solution of utilization of recycled irradiated high-density polyethylene (HDPE). A powder of recycled irradiated HDPE was used as filler into pure low-density polyethylene (LDPE) and tensile test was performed. Materials were mixed together in concentrations from 10 to 60 % of the filler and injection molded to produce test specimens. Tensile test was performed at ambient – 23°C temperatures. Observed results were Young´s Modulus and Ultimate Tensile Strength to determine tensile behavior and to describe influence of the filler on this behavior. Findings from this research work could help to solve problems with the utilization of irradiation cross-linked PE in the end of its lifetime.

***Keywords*—**Recyclation, HDPE, LDPE, tensile test, irradiation, properties.

I. Introduction

POLYMERS rank among a construction materials which find use in industry branches the most. The advantage of polymers is a low weight together with the excellent mechanical properties, very good chemical resistance and other properties, which assign them for various applications. Disadvantage is mainly low temperature stability which significantly reduces usage of these polymers.

Every property improvement especially temperature stability helps to increase application possibilities. In addition, property modifications of standard polymers, which are relatively cheap products, give them advantage for another usage. One of the possibilities of improvement of polymers' properties is radiation cross-linking.

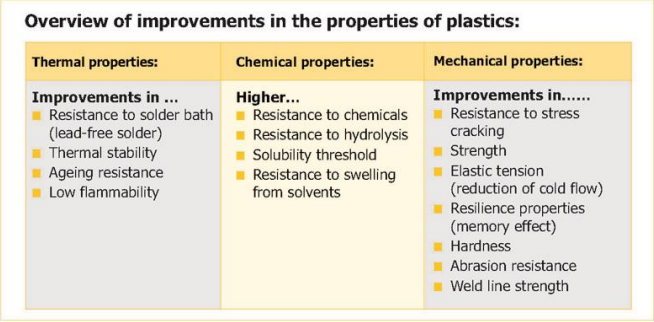

Fig. 1 Improved properties [2]

Cross-linking, i.e. creation of a network in polymer structure occurs mainly in the amorphous regions of polymers. The degree of cross-linking is dependent on the radiation dose and energy [1-10].

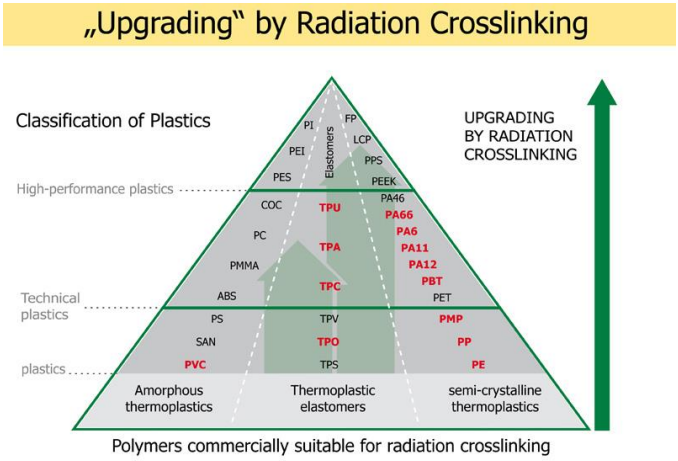

Fig. 2 Pyramid of polymers [2]

Cross-linking is the intermolecular bond formation of polymer chains. The mechanism of cross-linking involves the cleavage of a C-H bond on one polymer chain to form a hydrogen atom, followed by abstraction of a second hydrogen atom from a neighboring chain to produce molecular hydrogen. Then the two adjacent polymeric radicals combine to form a crosslink. The overall effect of cross-linking is that the molecular mass of the polymer

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steadily increases with radiation dose, leading to branched chains until a three-dimensional polymer network is formed when each polymer chain is linked to another chain [3, 4].

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 not 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). 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 accelerators, the required dosage can be applied within seconds, whereas several hours are required in the gamma radiation plant (Fig. 3). [1, 2]

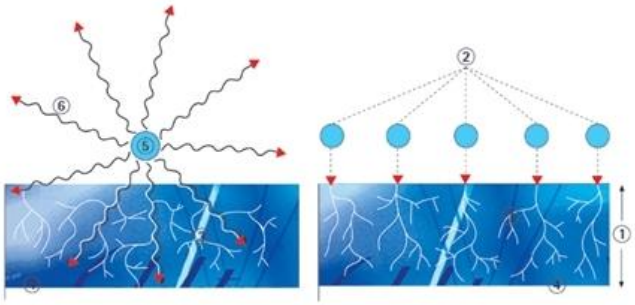

Fig. 3 Design of gamma rays (a) and electron rays (b),
1 – Penetration depth of an electron, 2 – Primary electron,
3 – Secondary electron, 4 – Irradiated material,
5 – Encapsulated Co – 60 Radiation source, 6 – Gamma rays [2]

Radiation cross-linking usually improves strength, reduces creep, contributes to chemical resistance improvement and in many cases improves tribological properties. Effect of radiation cross-linking significantly improves temperature stability. Because of that, materials which belong to group of standard polymers can be used in applications, which would be in term of temperature stability intended only to construction thermoplastic polymers.

In comparison with other construction / engineering materials, mainly metals; polymers including TPE, PE and PAs have limited levels of both mechanical and thermal properties. These limitations significantly reduce the applicability of polymers. Every improvement of these properties, of course, makes their applicability wider. Irradiation of thermoplastics is an important way to change their thermal properties. From the usage point-of-view, it is mainly the temperature stability which is a very important factor. Polymers can be irradiated in many forms, such as pellets and powder, films, extruded and molded parts or as wire and cable insulation [1]. Plastic parts suitable for radiation cross-linking are extruded products like tubes, pipes and profiles as well as injection-moulded parts.

II. MATERIAL AND METHODS

A. Material Preparation

Low-density polyethylene (LDPE), high-density polyethylene (HDPE) and LDPE with recycled irradiated HDPE as filler were tested.

LDPE was used as a polymer matrix due to its advantageous combination of low price, processing properties, rigidity and availability; moreover, one of the main areas of application of this material is compounding. Supplier of this material was The Dow Chemical Company, type 780E. This material was supplied in the form of granules.

Tab. 1 LDPE material properties [6]

DOW LDPE 780E	
Density	0.923 [g/cm^2]
Mass-Flow Rate (190°C, 2.16kg)	20 [g/10min]
Molding Shrinkage (Average)	1.9 [%]
Tensile Modulus	164 [MPa]
Tensile Stress at Break	10.5 [MPa]
Tensile Strain at Break	50 [%]
Tensile Impact Strength	286 [kJ/m^2]
Shore D Hardness	49
Vicat Softening Temperature	93 [°C]

Recycled HDPE material was provided in the form of tubes which served for floor heating. These tubes could not be remelted due to their modification by irradiation; therefore, they were used as active filler. Irradiation was performed by electron beams (beta radiation) with energy 10 MeV by total dose of 132kGy. This material was chosen for re-processing due to its growing usage for irradiation and thus increasing potential for recyclation after service life. Supplier of raw material was Slovnaft Petrochemicals, Inc., HDPE type TIPELIN PS 380-30/302.

Tab. 2 HDPE material properties [5]

Slovnaft TIPELIN PS 380-30/302	
Density	0.949 [g/cm^2]
Mass-Flow Rate (190°C, 5kg)	0.95 [g/10min]
Tensile Strength	31 [MPa]
Elongation at Break	1400 [%]
Flexural Modulus	750 [MPa]
Izod Impact Strength	13 [kJ/m^2]
Shore D Hardness	60.5
Vicat Softening Temperature	120 [°C]

Specimens' preparation was carried out in several steps.

Firstly those tubes were cleaned and shortened to the suitable length and crushed in the rotary cutter mill to grit (Fig.4).

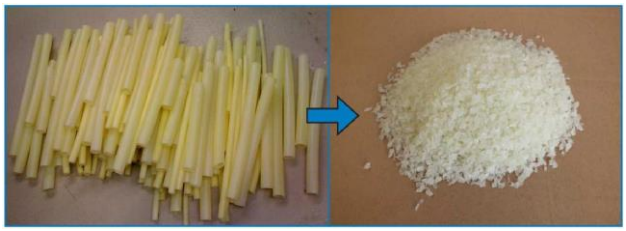

Fig. 4 Processed tubes

Particle size which leaves the mill varies between 3 to 5 mm. Diversity in shape and size is shown in Fig.5.

Fig.5 Particles shape and size

Thus prepared materials were then sent for grinding and resulting powder was used as filler/ polymer matrix. Before injection molding these materials underwent sieving to determine size of the particles. Sieving was carried out on the sieving machine where totally 200 g of powder was used. Sieving time was set to 30 minutes and amplitude of vibration was set to 90 mm. Measured data is shown in Fig.6.

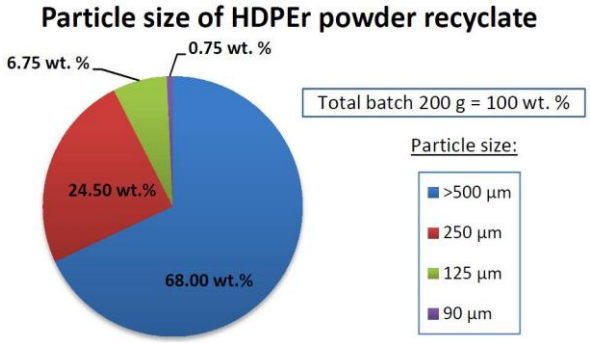

Fig.6 HDPE particle size

As can be seen from the chart above (Fig. 6) there were 68 wt. % of particles greater than 500 µm, 24.5 wt. % varied between 250 µm and 500 µm, 6.75 wt. % was between 125 µm and 250 µm and finally 0.75 wt. %was from 90 µm to 125 µm.

Thus prepared raw LDPE material was mixed together in concentrations from 10 % to 60 %. Mixing was carried out in a "home-made" laboratory pneumatic blender. The initial pressure under which an air was blown into the device was 7 bar, time of mixing was chosen to be 5 minutes.

Resulting compound was injection molded in injection molding machine ArburgAllrounder420C under process parameters shown in Tab. 3 and Tab. 4; nevertheless, with increasing concentration of filler these process parameters had to be slightly changed. Therefore at 50 % concentration of filler was injection pressure increased to 500 bar and holding pressure to 450 bar. At 60 % concentration of this filler were both these parameters raised to 550 bar due to deteriorated fluidity. To support this was MFR determined according to the standard ISO1133 (Fig. 7).

Tab. 3 Process parameters

Process parameters	
Injection Velocity	60 [mm/s]
Injection Pressure	450 [bar]
Injection Time	0.4 [s]
Cooling Time	30 [s]
Mold Temperature	40 [°C]
Feeding Length	27.5 [mm]
Pressure at V/P	400 [bar]
Point of V/P	10 [mm]
Clamping Force	950 [kN]
Feeding Time	2.8 [s]
Packing Phase	10.1 [s]
Packing	400 [bar]
Cycle Time	55.5 [s]

Tab. 4 Process temperature

Temperature Zones of Plasticization Unit	
Zone 1	135 [°C]
Zone 2	140 [°C]
Zone 3	150 [°C]
Zone 4	160 [°C]
Zone 5	180 [°C]
Temperature Under the Hopper	40 [°C]

Fig.7 Mass flow rate

Mass Flow Rate (MFR) decreased from 20.152 g/10min at non-filled pure LDPE to 0.796 g/10min at 60 % concentration of the filler. This equals to 96 % drop in compound fluidity. Both values are corresponding to the values from their datasheets and it can be seen that the more filler the lower fluidity; this finding justifies necessary changes in process settings.

Resulting test specimens had dimension and shape according to the standard ISO527 (Fig. 8 and Table 5).

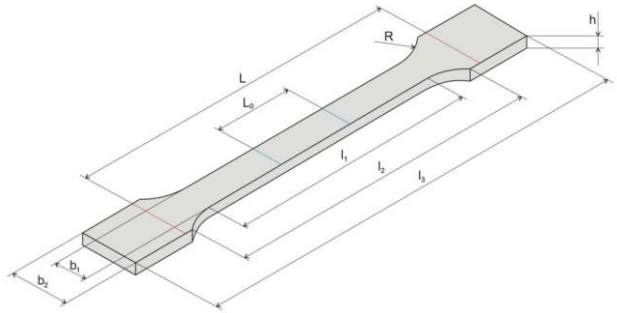

Fig. 8 Testing Specimen [28]

Tab. 5 Specimen dimensions [28]

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]

B. Used Methods for the Testing

The mechanical properties of selected polymers were measured. Tensile test was carried out on tensile test machine ZWICK 1456, according to standard CSN EN ISO 527-1, 527-2 with used rate 50mm/min. Test data was processed by Test Expert Standard software and modulus (E [MPa]) and tensile stress (σ_t [MPa]) were determined. The hardness was measured using a HPE – D Berreiss hardness tester type, and the Shore D Method. The shape and the dimensions of the testing samples were in accord with the CSN 621431 standard.

III. Results and Discussion

The mechanical behavior of low density polyethylene (LDPE) and high density polyethylene (HDPE), before and after irradiation was studied. Then the mechanical behavior of LDPE with recycled irradiated HDPE as filler was studied. For easier of evaluation of the measured data and the irradiated polymer with the non-irradiated one and the filled LDPE with the non-filled one were compared to describe influence of irradiation/filling on changes in mechanical behavior in percent.

A. *Tensile Test and Hardness*

Irradiation affects the mechanical properties of the LDPE, HDPE and LDPE with the filler were studied at the ambient temperature.

1) *Tensile Strength and E-modulus*

Irradiation affects the tensile strength of the LDPE studied at the ambient temperature (Fig. 9).

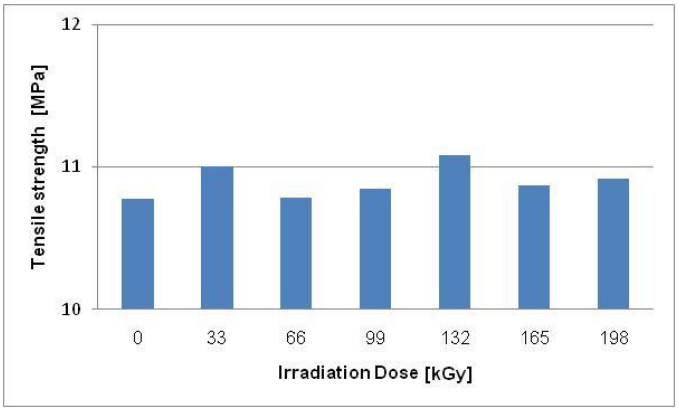

Fig. 9 LDPE Tensile strength at the temperature 23°C

At the ambient temperature tensile strength of both non-irradiated and irradiated LDPE is almost constant about 11MPa. The highest LDPE tensile strength with the dose of irradiation 132kGy was measured.

Irradiation affects the E-modulus of the LDPE studied at the ambient temperature (Fig. 10).

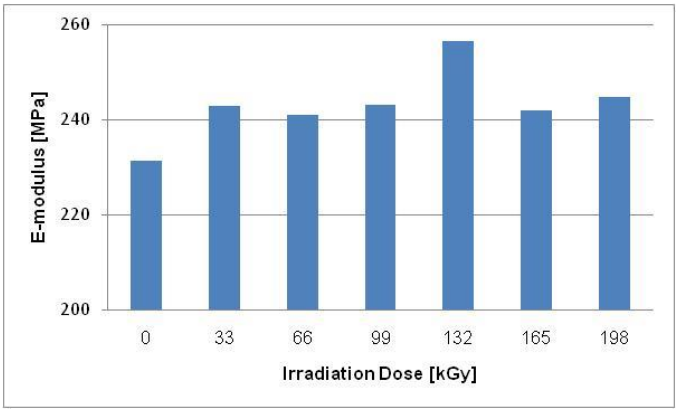

Fig. 10 LDPE E-modulus at the temperature 23°C

At the ambient temperature E-modulus of irradiation LDPE with a dosage of 132kGy, was increased about 25 MPa compared to non-irradiated LDPE.

As can be seen from Fig. 11 there is little influence of irradiation on tensile strength. The highest tensile strength was achieved at 132kGy irradiation dose; nevertheless, this increase is only 3% comparing to non-irradiated one.

Elastic modulus is more influenced by irradiation but again the change is not significant. The highest elastic modulus was achieved at 132kGy irradiation dose; the growth was 10% (Fig. 11).

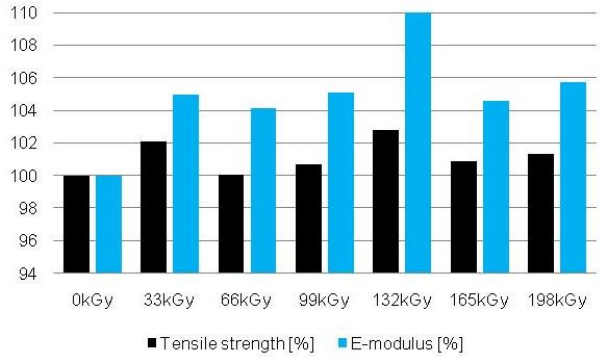

Fig. 11 Comparison of LDPE tensile behavior at 23°C

In the Fig. 12 can be seen irradiation affects the tensile strength of the HDPE studied at the ambient temperature.

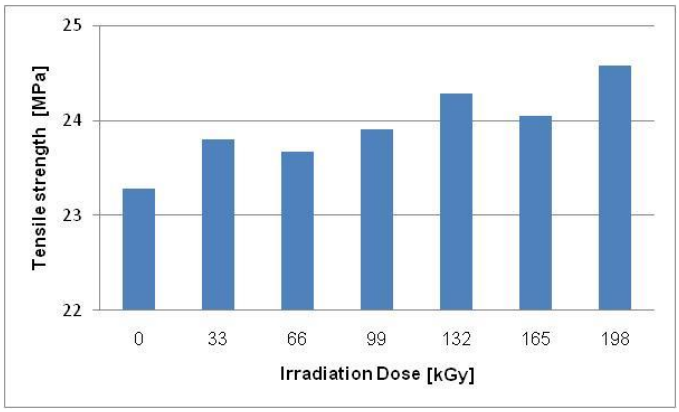

Fig. 12 HDPE Tensile strength at the temperature 23°C

At the ambient temperature tensile strength of irradiation HDPE with a dosage of 198kGy, was increased about 1,3 MPa compared to non-irradiated HDPE.

In the Fig. 13 can be seen irradiation affects the E-modulus of the HDPE studied at the ambient temperature.

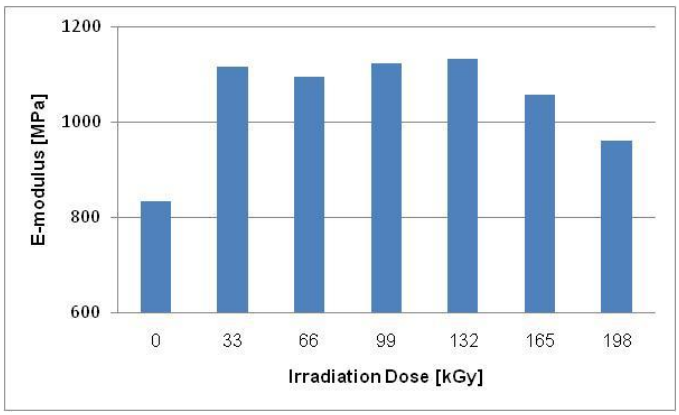

Fig. 13 HDPE E-modulus at the temperature 23°C

At the ambient temperature HDPE E-modulus increased up to 30kGy. It is stable up to 132kGy and then it begins to decrease.

In the Fig. 14 it is shown the dependence of irradiation dose on the change of tensile behavior of HDPE at 23°C.

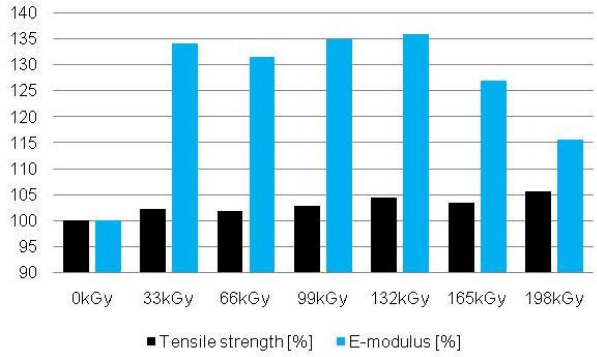

Fig. 14 Comparison of HDPE tensile behavior at 23°C

Similar trend was measured at HDPE (Fig. 14) as LDPE measured data. The tensile strength grown only by 5%; this was achieved at the highest irradiation dose – 198kGy. Elastic modulus increased significantly with irradiation dose; however, the value of irradiation itself had only little influence on elastic modulus. The highest elastic modulus was achieved at 132kGy irradiation dose – 136%.

Due to the higher nominal values of HDPE was this material used as filler into non-irradiated LDPE after its service life to investigate possible recyclation of such modified material.

In Fig. 15 can be seen that the higher level of filling the higher tensile strength.

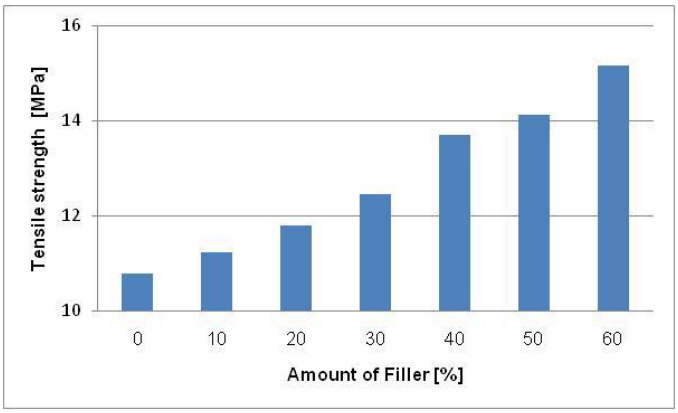

Fig. 15 Influence of filler on the LDPE tensile strength at 23°C

At the ambient temperature the tensile strength of filled LDPE with the dose of filling 60%, was increased about 4,2MPa compared to non-filled LDPE.

In Fig. 16 can be seen that the higher level of filling the higher E-modulus, it is the same trend as the trend of LDPE tensile strength (Fig. 15).

At the ambient temperature the E-modulus of filled LDPE with the dose of filling 60%, was increased about 200MPa compared to non-filled LDPE.

In Fig. 17 can be seen that the higher level of filling the higher elastic modulus as well as tensile strength. In case of elastic modulus the increase is up to 85% and in case of tensile strength it grown up to 40%.

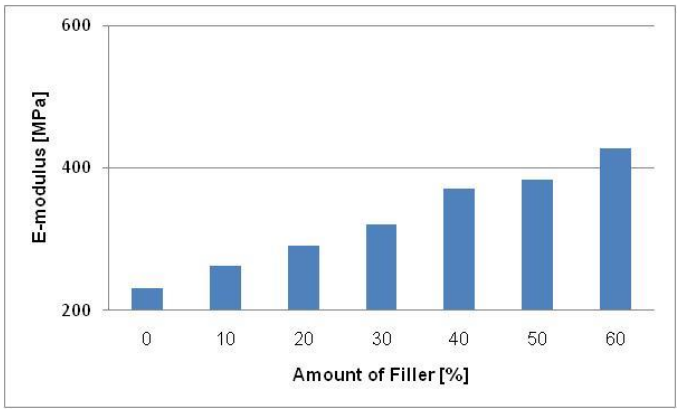

Fig. 16 Influence of filler on the LDPE E-modulus at 23°C

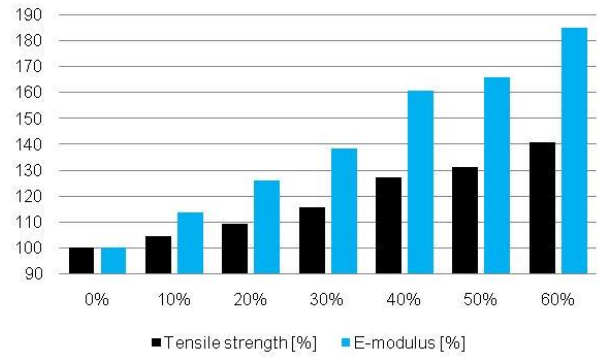

Fig. 17 Influence of filler on the LDPE tensile behavior

2) Hardness

Irradiation affects the hardness of LDPE, HDPE and LDPE with the filler were studied at the ambient temperature. In the Fig. 18 it is possible to see the changes of hardness of LDPE at the ambient temperature.

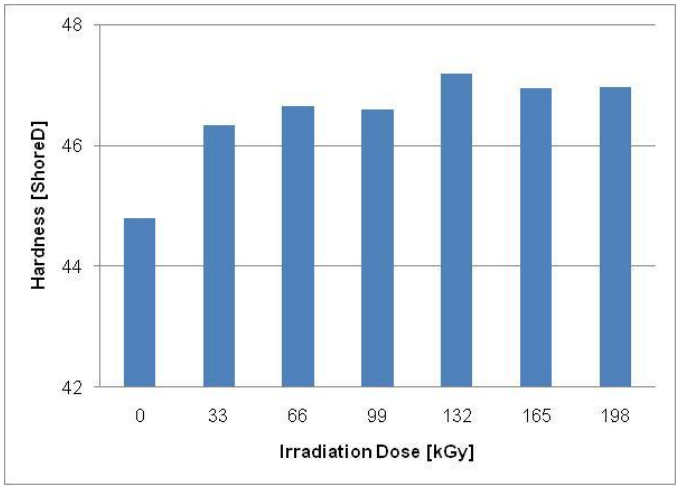

Fig. 18 LDPE Hardness at the temperature 23°C

No significant changes of hardness were found after the irradiation on the LDPE samples. A maximum increase of LDPE hardness (i.e. about 2%) was measured after the irradiation dosage of 132kGy.

In the Fig. 19 it is possible to see the changes of hardness of HDPE at the ambient temperature.

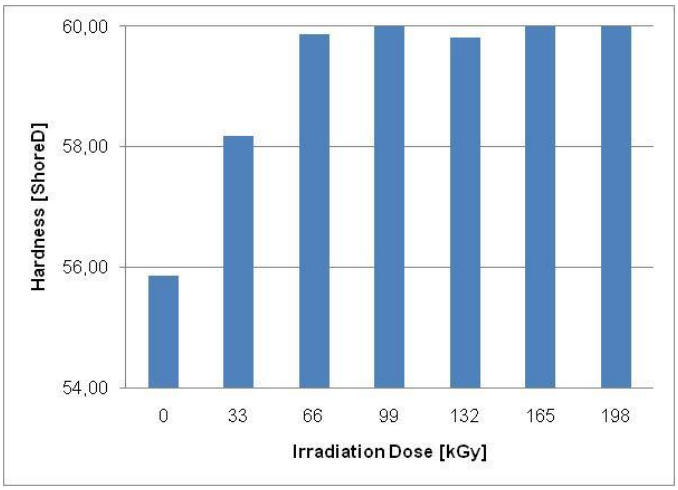

Fig. 19 HDPE Hardness at the temperature 23°C

At the ambient temperature Shore D hardness of irradiation HDPE with a dosage of 66kGy was increased about 4 Shore D compared to non-irradiated HDPE.

In the Fig. 20 is shown the dependence of irradiation dose on the change of hardness for LDPE and HDPE at 23°C.

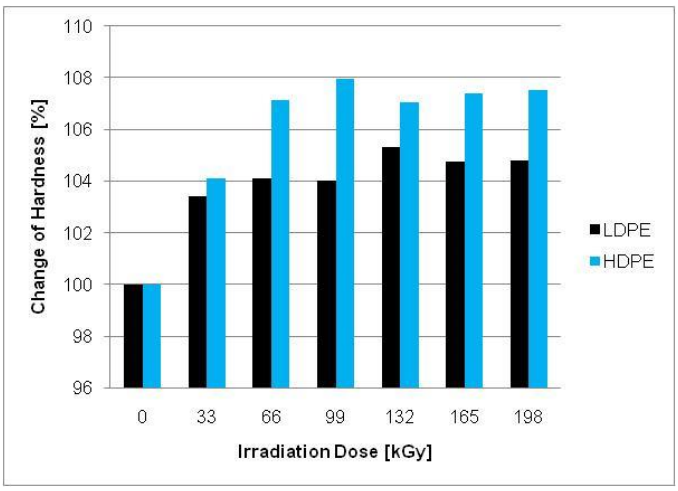

Fig. 20 Comparison of hardness of LDPE and HDPE at 23°C

No significant changes of hardness were found after the irradiation on the LDPE and HDPE sample. A maximum increase of HDPE hardness (i.e. about 8%) was measured after the irradiation dosage of 99kGy. Higher doses of irradiation had no significant effect on the Shore D Hardness.

In the Fig. 21 it is possible to see influence of filler on the LDPE hardness at the ambient temperature.

At the ambient temperature the hardness of filled LDPE with the dose of filling 60%, was increased about 9,3 Shore D compared to non-filled LDPE.

Results show (Fig. 22) steady increase up to 20% at the highest level of filling. This confirms positive effect of usage irradiated material as filler into non-irradiated one.

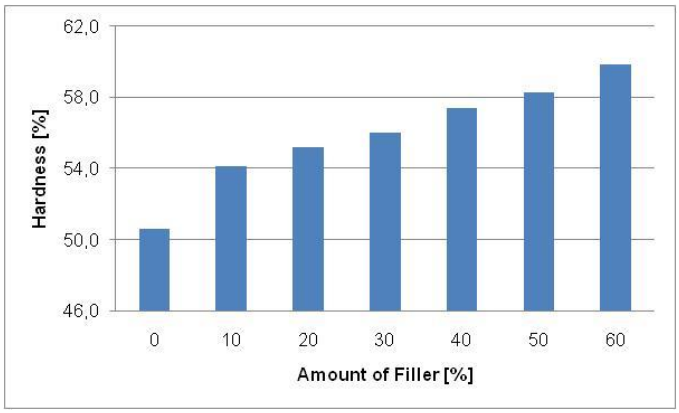

Fig. 21 Influence of filler on the LDPE hardness at 23°C

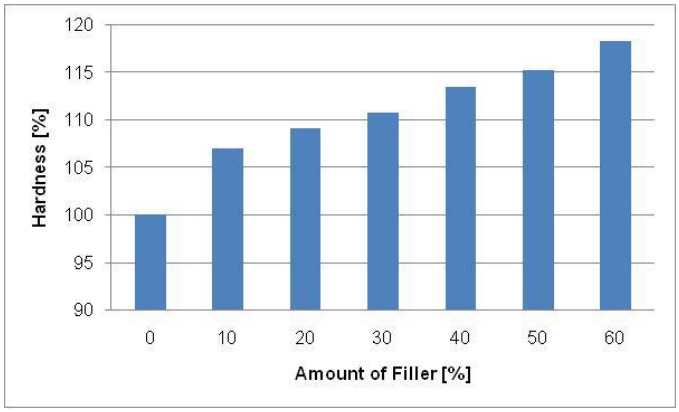

Fig. 22 Influence of filler on the LDPE hardness in percentage

IV. Conclusion

Plastics are by far the largest group of polymeric materials being processed by electron beam irradiation. Cross-linking of polyolefins, PVC, polyesters, polyurethanes, fluoropolymers and fiber-reinforced composites are a common practice. Radiation cross-linking of PE requires considerably less overall energy and space, and is faster, more efficient and more environmentally acceptable. The disadvantage of electron beam cross-linking is a more or less nonuniform dose distribution. This can happen, particularly in thicker objects, due to intrinsic dose-depth profiles of electron beams. Another problem can be a nonuniformity of rotation of cylindrical objects as they traverse a scanned electron beam. However, the mechanical properties often depend on the mean cross-link density. [1]

Influence of irradiation on tensile behavior was investigated. Two types of materials were tested – HDPE and LDPE. Results indicate insignificant changes in this material property in case of LDPE. In case of HDPE there is limited influence on tensile strength; however, elastic modulus grown sharply. Usage of irradiated HDPE after its service life was also tested. Pipes made of this material were processed to powder which was used as filler into non-irradiated LDPE. Results show positive influence on all measured variables which gives us possible solution of

recyclation of irradiated materials. However further research is necessary to fully understand this problem.

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