Material properties´ comparison of PA6/HDPEx blends

Jan Navratil, Miroslav Manas, Michal Stanek, David Manas, Martin Bednarik, and Ales Mizera

Abstract—Irradiation of thermoplastics is a very spread material modification for improving their properties, yet little research has been done to investigate possible utilization of such modified materials after the end of their lifetime. This research paper tries to give possible solution of using this material as a filler into some other one. The emphasis is put on utilization of irradiated high-density polyethylene (HDPEx) which has been blended with non-modified polyamide 6 (PA6). Two concentrations of tested blends were prepared (10 and 30 %) when raw PA6 matrix was in a form of granules and raw HDPEx waste in a form of grit. Three mechanical properties tests were performed in order to get the most complex results of the resulting mechanical behavior. Tensile properties were tested at two temperatures and at both of them was a decline observed. Elastic modulus decreased from 3591 to 1815 MPa at 24 °C and from 533 to 294 at 80 °C. Impact toughness was investigated via impact charpy notched test where the results varied greatly. Last observed property was hardness where it slightly declined from 77.8 to 74.4 Shore D. All results show that HDPEx waste can be processed as a filler; however when mixed with PA6 there is significant loss of original PA6 properties.

Keywords—HDPE, irradiation, PA6, radiation crosslinking, recycling, material properties.

I. INTRODUCTION

KNOWLEDGE of polymer irradiation has led to an increasing usage of cheap commodity plastics in the areas where it was unthinkable before. Original purpose was sterilization but since positive effect on plastics was discovered it is used in plastic industry as well. Irradiation causes crosslinking or degradation in the structure of exposed polymer. Those polymers which tend to crosslinking (Fig. 1) have significantly improved mechanical, thermal and chemical properties [1]-[18].

Crosslinking, i.e. formation of a 3D network in a polymer structure occurs mainly in the amorphous region of the polymer. The degree of crosslinking depends on the radiation dose and radiation energy. From chemical point of view is crosslinking intermolecular bond formation of polymer chains. The mechanism of crosslinking involves the cleavage of the C-H bond on one polymer chain to form a hydrogen atom H followed by abstraction of a second hydrogen from another polymer chain to produce a hydrogen molecule H_2. Then the two adjacent polymer radicals combine and form a crosslink (Fig. 2) [1]-[18], [23]-[47].

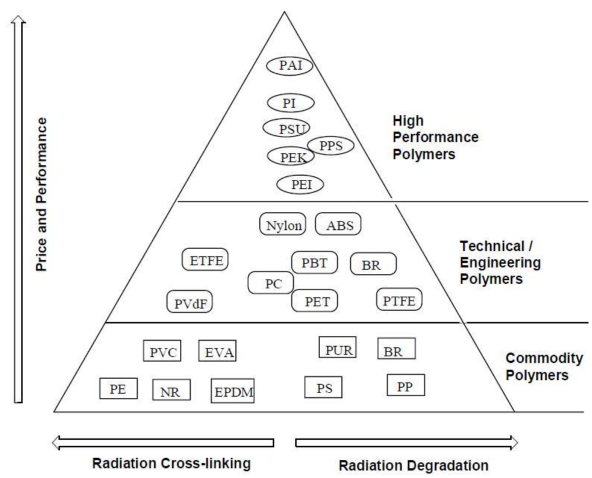

Fig. 1 Material classification [3]

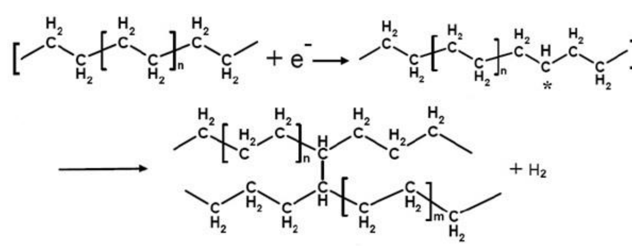

Fig. 2 Crosslinking mechanism of polyethylene [4]

The source of radiation may be an electron beam accelerator emitting beta radiation or a radioactive isotope such as Cobalt-60 emitting gamma radiation [49]-[64].

Electron beam accelerator is the most used radiation source which emits monoenergetic beam. The highest dose is just below the surface of the irradiated material and falls rapidly at higher penetration depths, therefore it is not suitable for

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irradiating thick materials. The energy range of electron beams used in irradiation of polymers varies between 0.15 and 10 MeV. Irradiation takes place at ambient conditions and on the final product hence processability is not influenced. The principle of producing beta radiation is simple. The electrons are emitted in vacuum by a heated cathode and accelerated in the electrostatic field applied between cathode and anode. Acceleration takes place from the cathode, which is connected to a negative high-voltage potential, to the grounded accelerator window as anode. Usually an electron optical system is used to focus the accelerated electrons to the accelerator window plane [23]-[47], [49]-[64].

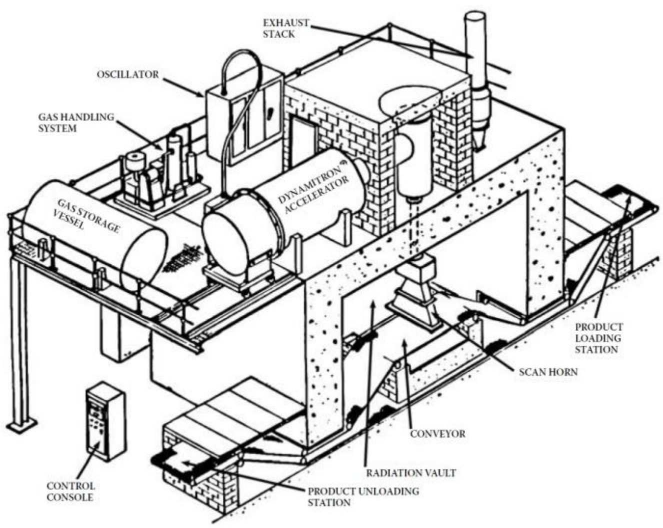

Fig. 3 Typical electron beam accelerator facility [11]

II. EXPERIMENT

This experiment deals with preparation of PA6/HDPEx blends and thus with determination of possible utilization of irradiated HDPE waste. Resulting injection molded blends underwent series of tests to investigate their physical properties.

A. Materials

Combination of two materials was tested – neat polyamide 6 (PA6) and waste of irradiated high-density polyethylene (HDPE). HPDEx waste was used as a filler into the PA6 in two concentrations (10 and 30 %).

PA6 has, as an engineering plastics, very good mechanical properties and therefore it was chosen as a polymer matrix. Producer of this material was Frisetta, type FRIANYL B63 V0. This material was provided in form of granules, its basic properties are shown in table I.

Table I PA6 material properties [20]

FRIANYL B63 V0	
Density	1.2 [g/cm^3]
Water Absorption (23 °C)	2-3 [%]
Tensile Modulus	3500 [MPa]
Tensile Strength	60 [MPa]
Tensile Elongation at Break	3 [%]
Charpy Impact (notched 23 °C)	5 [kJ/m^2]

HPDEx waste was provided in form of pipes, which were crushed into a grit of 3 to 5 mm particle size. Pipes were originally irradiated by beta radiation with the energy 10 MeV by the total dose of 165 kGy. Irradiation caused irreversible creation of 3D network in the HDPE structure and therefore this material could not be remelted repeatedly and had to be used as a filler. Properties of the original HDPE are shown in table II; however properties of the used HDPEx slightly differ due to the irradiation. Producer of the original material was Slovnaft, type TIPELIN 6300B.

Table II HDPE material properties [19]

TIPELIN 6300B	
Density	0.954 [g/cm^3]
Melt Flow Rate (190 °C / 2.16 kg)	0.3 [g/10 min]
Vicat Softening Temperature	126 [°C]
Tensile Strength	29 [MPa]
Shore D Hardness	65 [-]
IZOD Impact Strength (notched 23 °C)	9 [kJ/m^2]

B. Specimens´ Preparation

Raw materials were mixed together in a laboratory pneumatic blender in concentrations 10 and 30 % of the filler. Resulting mixtures were dried for five hours at 80 °C and then injection molded in injection molding machine Arburg Allrounder 470H. Specimen shape for tensile behavior testing was according to the ISO 527 standard (Fig. 4), specimen shape for hardness/impact toughness testing was according to the ISO 868/179 standard (Fig. 5). Processing parameters were the same for all the concentration which might suggest minimal influence on processability of the resulting mixtures.

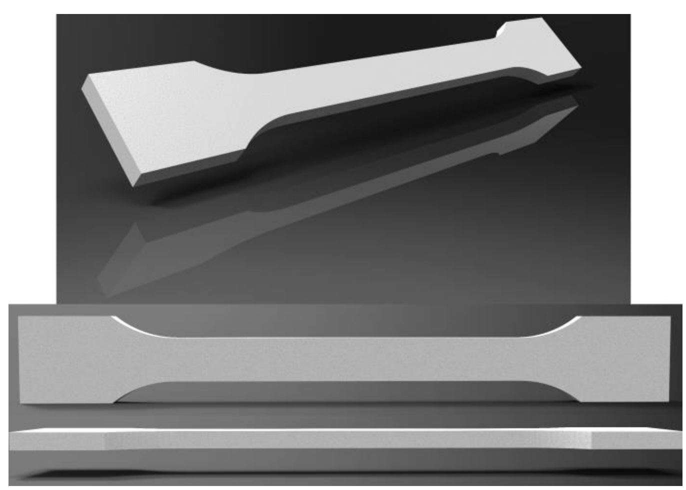

Fig. 4 Tensile behavior specimen [21]

Fig. 5 Hardness/impact toughness behavior specimen [22]

III. Results

Three material characteristics were tested in order to describe mechanical behavior in the most complex way. Tensile test was chosen to describe toughness and strength under static load, hardness was chosen to examine surface resistivity and finally impact toughness was chosen to describe material behavior under dynamic load.

A. *Tensile test*

Tensile test was performed at ambient temperature conditions (24 °C) and in the temperature chamber at elevated temperature conditions (80 °C).

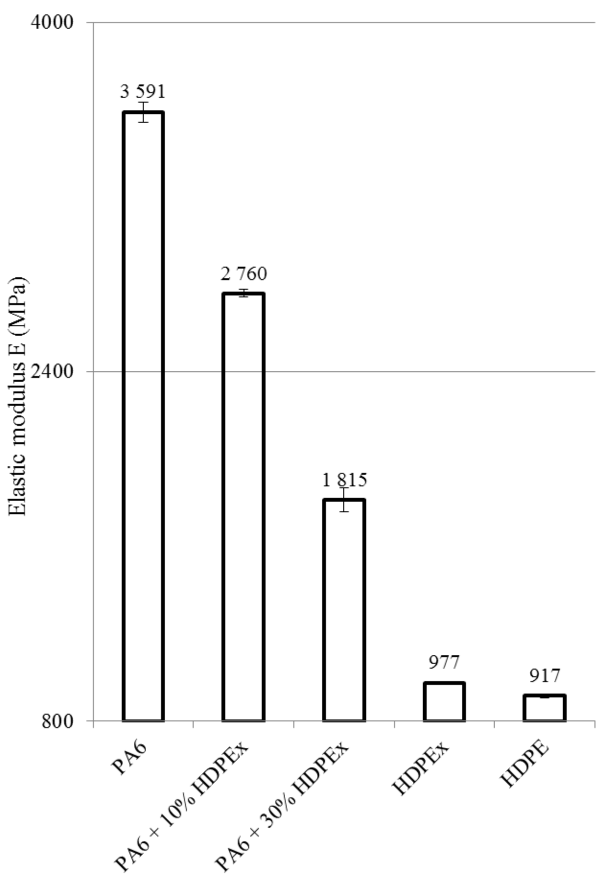

Fig. 6 Elastic modulus at 24 °C

First observed characteristics was elastic modulus and as can be seen from Fig. 6 it significantly decreased with increasing amount of the filling. Reference value of non-modified PA6 was not achieved. First concentration of filling (10 %) resulted in 23 % drop from the reference value. Second concentration (30 %) resulted in almost 50 % drop. This suggests that there was not very good adhesion between filler particles and polymer matrix. However despite this significant drop from reference value both concentration have still higher elastic modulus than irradiated (977 MPa) and non-irradiated (917 MPa) HDPEs. By comparing these HDPEs can be seen that irradiation has little influence on the elastic modulus.

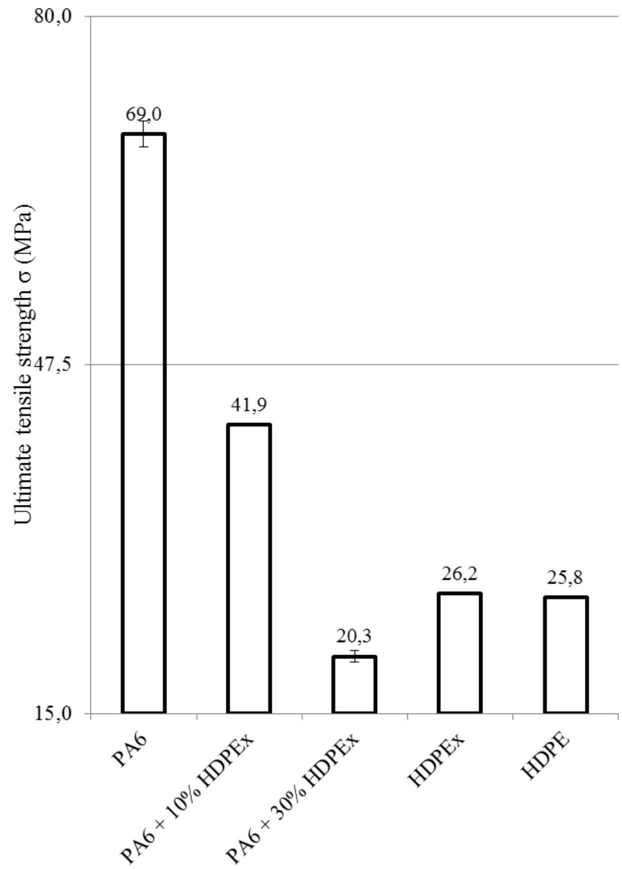

Fig. 7 Ultimate tensile strength at 24 °C

Ultimate tensile strength results show similar trend as the elastic modulus (Fig. 7); however the drop is even more significant. First concentration dropped from 69 to 41.9 MPa which represents 40 % drop. Second concentration decreased to 20.3 MPa which represents 70 % drop. This shows that our presumption of bad adhesion is confirmed and thus filling PA6 is not suitable for improving tensile behavior of the resulting material. Ultimate tensile strength of non-filled yet modified HDPEx is 26.2 MPa which is fully comparable with non-filled and non-modified HDPE (25.8 MPa). This again confirms little influence of radiation crosslinking on resulting tensile behavior.

Nominal strain represents elasticity of the material and as is depicted in Fig. 8 there is a slight loss of elasticity with increasing amount of the filler. Reference strain of PA6 was 5.126 whereas the first concentration has strain 3.6 which is 30 % difference. Second concentration has even lower elasticity (3.003). This result is not in correlation with previous two results because nominal strain, i.e. elasticity should increase with decreasing elastic modulus/tensile strength; however according to our results it decreases as well. It may be caused again by the little adhesion between both materials which results in delamination of specimens. Nominal strain of both HDPEs is according to the expectations. Slightly higher strain of non-modified HDPE is a result of slightly lower elastic modulus/tensile strain than HDPEx.

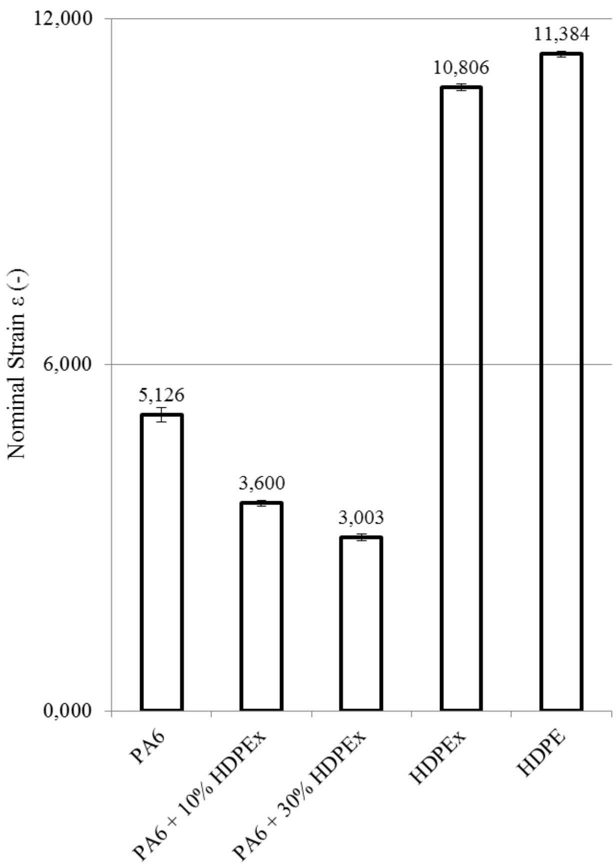

Fig. 8 Nominal strain at 24 °C

Results of tensile behavior measurements at ambient temperature conditions show that adding waste of irradiated HDPE in the PA6 does not improve final tensile mechanical properties; nevertheless there is an improvement comparing blends with both, modified and non-modified HDPE.

Elevated temperature of 80 °C was chosen due to the original purpose of provided HDPEx pipes. Those were used for floor heating with working temperature up to 80 °C. All specimens were conditioned at this temperature for at least 30 minutes before each measurement in order to achieve uniform temperature in the whole cross-section.

Elastic modulus was reduced from 533 MPa to 507 MPa at the first blend (10 %) as can be seen in Fig. 9. This reduction is significantly lower than at ambient temperature conditions, it represents only 6 % reduction which might mean that intermolecular forces between the matrix and filler are stronger at higher temperatures. Second blend with 30 % of the filler dropped to 294 MPa which is similar decrease to the ambient temperature result (45 % drop). Comparably to the elastic modulus measured at ambient temperature conditions, measured data of all concentrations are higher than both HDPEs. Irradiated HDPE has elastic modulus 204 MPa and non-irradiated HDPE has elastic modulus 169 MPa. This also shows that influence of irradiation is higher at increased temperature. Values of all measurement are at least four times lower than those measured at ambient temperature conditions and standard deviation of measurement is on the other hand much higher.

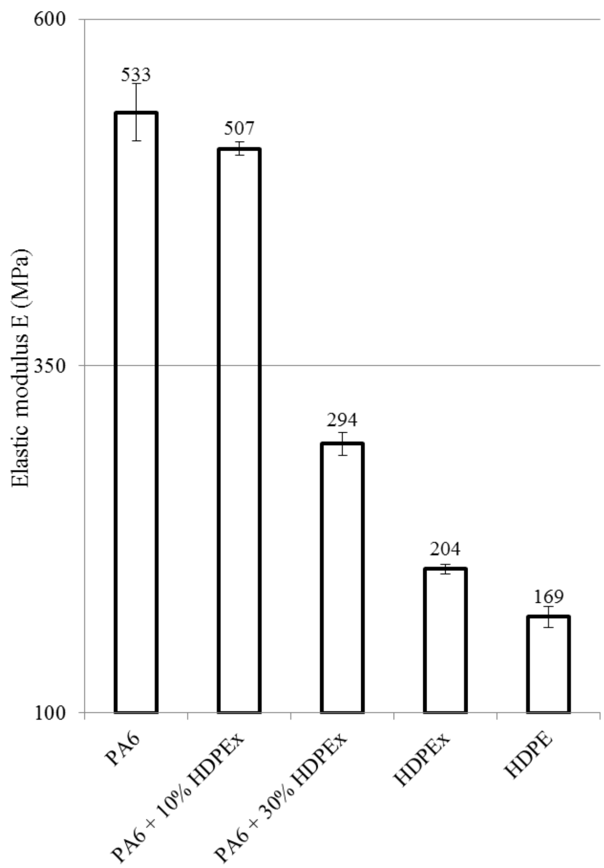

Fig. 9 Elastic modulus at 80 °C

Ultimate tensile strength results have again similar trend as elastic modulus (Fig. 10). There is unlike elastic modulus a significant drop in this material property even at the lowest concentration of filling where the difference between the non-modified reference PA6 and the first concentration of filling is 51 %. Another addition of the filler resulted in 72 % drop comparing it with the reference value. Results of both HDPEs are very similar and the difference between them is negligible. Influence of irradiation on measured material strength is thus very limited. Nominal values of ultimate tensile strength are at this elevated temperature not so significantly lower in comparison with results measured at ambient temperature.

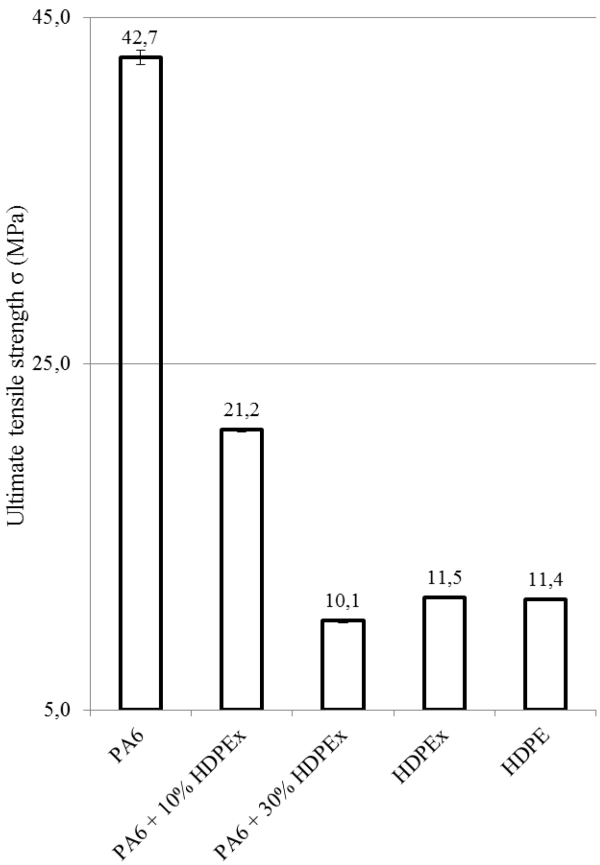

Fig. 10 Ultimate tensile strength at 80 °C

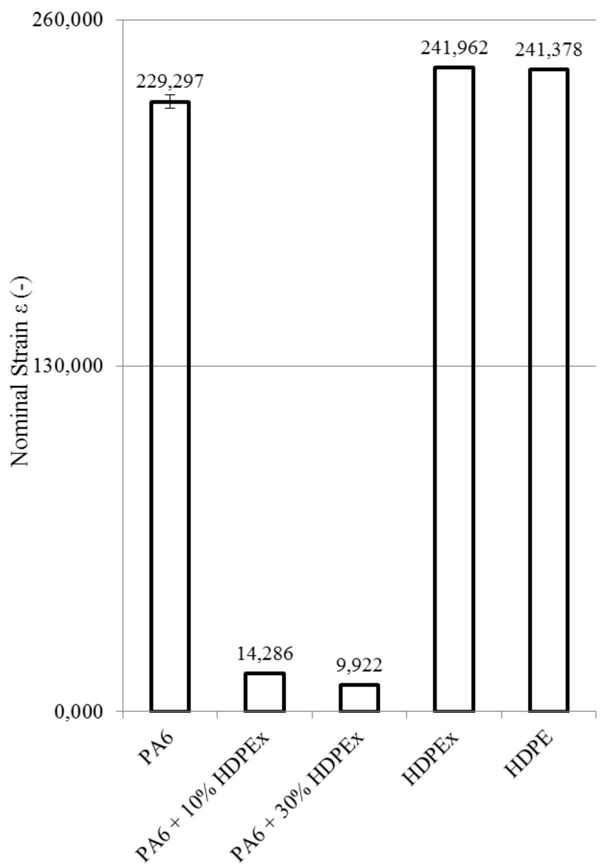

Fig. 11 Nominal strain at 80 °C

Nominal strain of the reference PA6 hugely increased at elevated temperature in comparison with ambient temperature result. It increased from 5.126 to 229.297 which shows that elevated temperature has huge effect on material elasticity. PA6/HDPEx blends were very little influenced by the temperature on the other hand. Their nominal strain increased from 3.6 to 14.286 at the first concentration and from 3.003 to 9.922 at the second concentration. By comparing reference PA6 and its blends can be seen that there is a big difference between them. The difference between PA6 and the first concentration is 215.011 which represents 94 % drop. The second concentration decreased by additional 4.364 which is additional 31 % drop. Nominal strain of both HDPEs also significantly grew at elevated temperature up to almost 242 which is a result of weakened intermolecular forces at elevated temperature. Influence of irradiation on nominal strain is again very little.

Results of tensile behavior measurements at elevated temperature conditions show similarly to ambient temperature that adding waste of irradiated HDPE in the PA6 does not improve final tensile mechanical properties.

B. *Impact toughness test*

Impact toughness test is one of the tests to determine dynamic material properties.

Charpy notched impact test was chosen to evaluated dynamic behavior. Notch size and shape was according to the CSN EN ISO 179 (type C). Evaluated variable was impact toughness Am (kJ/m^2) which can be seen in Fig. 12. This variable describes impact material toughness up to the maximum force F_M necessary for breaking the specimen.

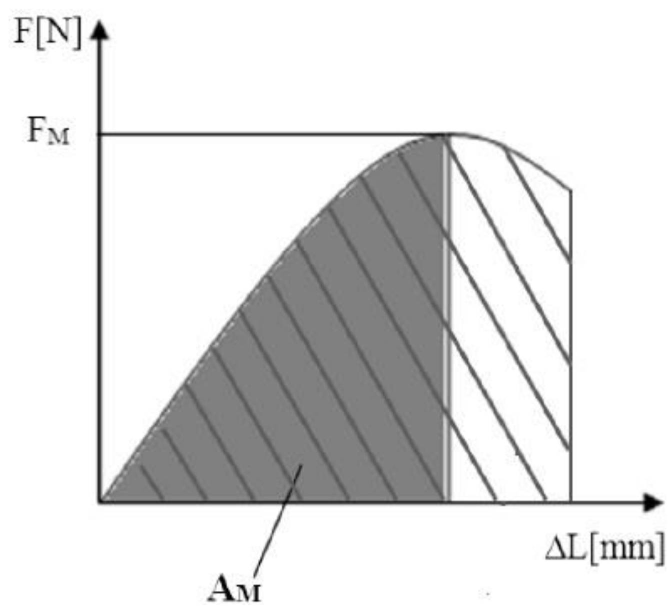

Fig. 12 Evaluated variable [48]

Measurement was carried out at standard conditions, weight of the hammer was 2.192 kg, radius of the arm was 0.5 m, impact angle was 40 °, impact energy was 2.51 J and impact velocity was 1.51 $m.s^{-1}$.

As can be seen in Fig. 13 impact toughness of the reference PA6 was slightly higher than stated in material list (Table I) which might be caused by different processing parameters. First concentration of filling resulted in significant increase in impact toughness. It grew from 7.37 to 17.46 kJ/m^2 which represents 137 % increase. Second concentration of filling had impact toughness 8.73 kJ/m^2 which is in comparison with the reference value only 18 % increase. This unusual behavior can be just an anomaly because this test is very sensitive on proper homogeneity and particle dispersion of the tested material hence more measurement would have to be necessary to diminish this error as much as possible. Impact toughness of irradiated HDPE is higher than non-modified HDPE which is caused by creation of the intermolecular bond in the structure of irradiated HDPE. Irradiated materials have in general higher impact toughness because crosslinking makes them more ductile.

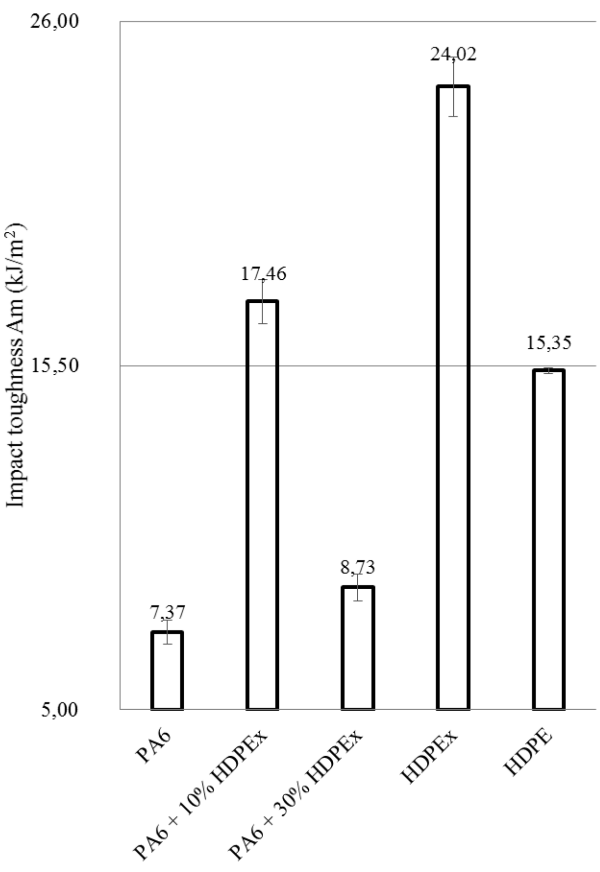

Fig. 13 Impact toughness test

C. *Hardness test*

Shore D hardness of the tested blends gradually decrease with increasing amount of the filling. Difference between the reference value (77.8 shore D) and the first concentration (77.5 shore D) is almost negligible. Difference between the reference value and the second concentration is higher (77.8 vs. 74.4); however it represents only 4 %. According to this result it can be stated that there is not significant influence of the filler on resulting shore D hardness. Hardness of the irradiated HDPE is higher in comparison with non-modified HDPE. The difference is 2 % which is also almost negligible; however in general irradiation improves material hardness.

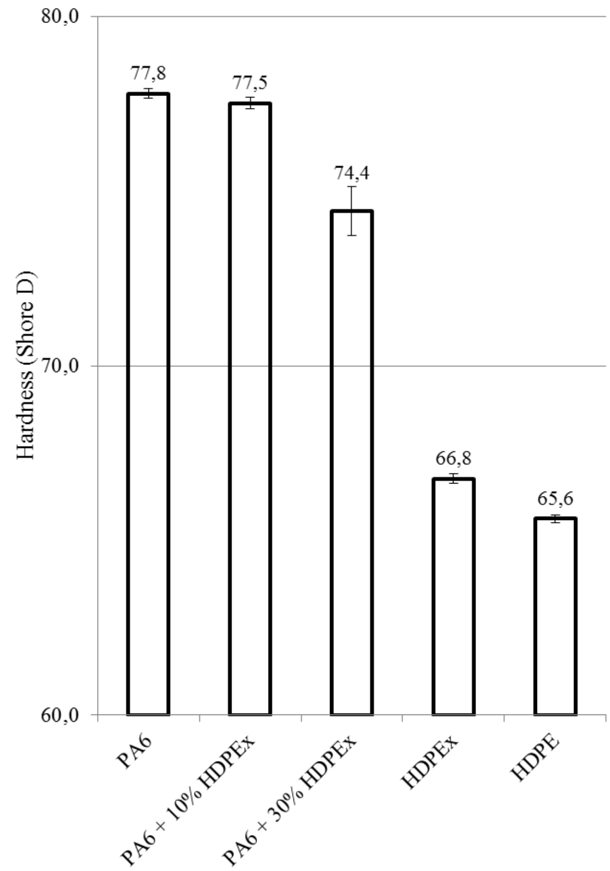

Fig. 14 Hardness test

IV. Conclusion

The main aim of this research paper was to determine possible utilization of HDPE modified by radiation crosslinking after its service life. This paper dealt with using grit of recycled HDPEx as a filler into granules of non-modified PA6. Two concentrations of proposed blends were prepared and testing specimens were injection molded. Mechanical properties of the resulting blends were tested when tensile test, charpy impact test and shore D hardness test were chosen to describe mechanical behavior the most. In case of tensile test testing was carried out at two temperature conditions (ambient and elevated) and three material characteristics were compared – elastic modulus, ultimate tensile strength and nominal strain. Results show that with increasing concentration of the filler decrease all these properties in case of comparing it with non-modified PA6. But when comparing it with non-modified or irradiated HDPE the values are higher (except nominal strain of both HDPEs). Impact toughness showed instable results most probably caused by non-homogeneous dispersion of filling particles. Shore D hardness was not influenced significantly. Therefore it may be concluded that it is possible to use such modified material as the filler; however it has to be carefully chosen what the final application will be and what material characteristics are important.

This research paper continues with investigating possible utilization of irradiated HDPE after its service life. More material combinations have to be tested to make final conclusion and to begin with industrial application of proposed solution.

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