

# Microhardness of HDPE influenced by Beta Irradiation

M. Ovsik, D. Manas, M. Manas, M. Stanek, K. Kyas, M. Bednarik and A. Mizera

**Abstract**—Hard surface layers of polymer materials, especially HDPE, can be formed by chemical or physical process. One of the physical methods modifying the surface layer is radiation cross-linking. Radiation doses used were 0, 33, 66, 99, 132, 165 and 199 kGy for HDPE. Individual radiation doses caused structural and micromechanical changes which have a significant effect on the final properties of the HDPE tested. Radiation doses cause changes in the surface layer which make the values of some material parameters rise. The improvement of micromechanical properties was measured by an instrumented microhardness test.

**Keywords**— Crosslinking, High Density Polyethylene (HDPE), irradiation, microhardness.

## I. INTRODUCTION

A linear polymer, High Density Polyethylene (HDPE) is prepared from ethylene by a catalytic process. The absence of branching results in a more closely packed structure with a higher density and somewhat higher chemical resistance than LDPE. HDPE is also somewhat harder and more opaque and it can withstand rather higher temperatures (120° Celsius for short periods, 110° Celsius continuously) [1], [4].

The irradiation cross-linking of thermoplastic materials via electron beam or cobalt 60 (gamma rays) proceeds in a proceeding separately after the processing. The cross-linking level can be adjusted by the irradiation dosage and often by means of a cross-linking booster.

The main difference between  $\beta$ - and  $\gamma$ - rays is in their different abilities of penetrating the irradiated material.  $\gamma$ - rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons.

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Due to electron accelerators the required dose can be applied within seconds, whereas several hours are required in the  $\gamma$ -radiation plant. The electron accelerator operates on the principle of the Braun tube, whereby a hot cathode is heated in vacuum to such a degree that electrons are released [2], [3], [15], [16].

Simultaneously, high voltage is generated in a pressure vessel filled with insulating gas. The released electrons are accelerated in this vessel and made to fan out by means of a magnetic field, giving rise to a radiation field. The accelerated electrons emerge via a window (Titanium foil which occludes the vacuum) and are projected onto the product [3], [7], [10].

Cobalt 60 serves as the source of radiation in the gamma radiation plant. Many of these radiation sources are arranged in a frame in such a way that the radiation field is as uniform as possible. The palleted products are conveyed through the radiation field. The radiation dose is applied gradually, that is to say, in several stages, whereby the palleted products are conveyed around the Co – 60 radiation sources several times. This process also allows the application of different radiation doses from one product type to another. The dimensional stability, strength, chemical resistance and wear of polymers can be improved by irradiation. Irradiation cross-linking normally creates higher strength as well as reduced creep under load if the application temperature is above the glass transition temperature ( $T_g$ ) and below the former melting point. Irradiation cross-linking leads to a huge improvement in resistance to most of the chemicals and it often leads to the improvement of the wear behaviour [3], [4].

The thermoplastics which are used for production of various types of products have very different properties. Standard polymers which are easy obtainable with favourable price conditions belong to the main class. The disadvantage of standard polymers is limited both by mechanical and thermal properties. The group of standard polymers is the most considerable one and its share in the production of all polymers is as high as 90%.

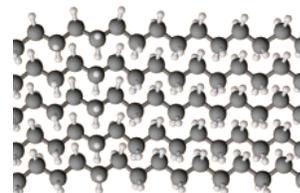


Fig. 1 Molecule structure of HDPE

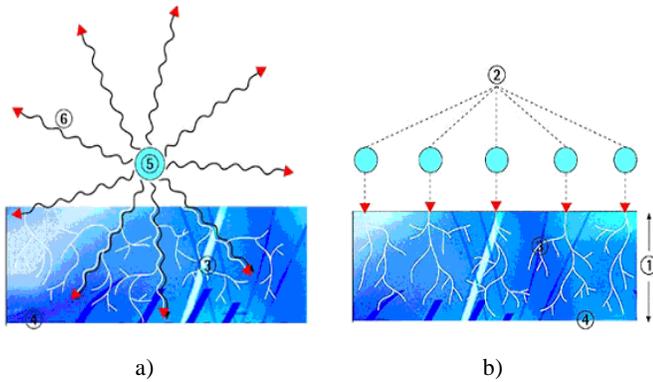


Fig. 2 Design of Gamma rays (a) and Electron rays (b)

- a) 3 – secondary electrons, 4 – irradiated material, 5 – encapsulated Co – 60 radiation source, 6 – Gamma rays  
 b) 1 – penetration depth of electron, 2 – primary electron, 3 – secondary electron, 4 – irradiated material

The engineering polymers are a very important group of polymers which offer much better properties in comparison to those of standard polymers. Both mechanical and thermal properties are much better than in case of standard polymers. The production of these types of polymers takes less than 1 % of all polymers [1], [6].

High performance polymers have the best mechanical and thermal properties but the share in production and use of all polymers is less than 1%.

The present experimental work deals with the influence of beta irradiation on the microhardness of HDPE.

## II. EXPERIMENTAL

### A. Irradiation

For this experiment High Density Polyethylene HDPE DOW – HDPE 25055E, DOW - Chemical company, USA (unfilled, HDPE ) was used. The prepared specimens were irradiated with doses of 0, 33, 66, 99, 132, 165 and 199 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany.

### B. Injection molding

The samples were made using the injection molding technology on the injection moulding machine Arburg Allrounder 420C. Processing temperature 200–240 °C, mold temperature 40 °C, injection pressure 80 MPa, injection rate 60 mm/s.

### C. Micro-hardness according to Vickers

Test of hardness according to Vickers is prescribed by European standard CSN EN ISO 6507-1.

The penetrating body – made of diamond shaped as a regular tetragonal pyramid with the square base and with preset vertex angle ( $136^\circ$ ) between opposite walls – is pushed against the surface of testing body. Then, the diagonal size of the dint left after load removal is measured (Fig. 2).

Vickers' microhardness is then expressed as the ratio of the testing load applied to dint area in form of regular tetragonal

pyramid with square base and the vertex angle equal to the angle of penetrating body ( $136^\circ$ ).

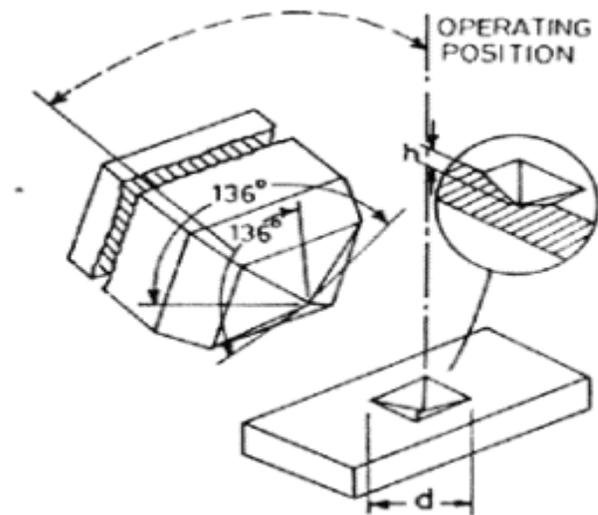


Fig. 3 The basic principle of hardness testing according to Vickers

### D. Instrumented microhardness tests

Instrumented microhardness tests were done using a Micro Combi Tester, CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1. Load and unload speed was 2 N/min. After a holding time of 90 s at maximum loads 0.5 N, 1 N and 5 N the specimens were unloaded.



Fig. 4 Micro-combi tester

The indentation hardness HIT was calculated as maximum load to the projected area of the hardness impression according to:

$$H_{IT} = \frac{F_{max}}{A_p} \quad \text{with} \quad h_c = h_{max} - \varepsilon \frac{F_{max}}{S} \quad (2)$$

where  $h_{max}$  is the indentation depth at  $F_{max}$ ,  $h_c$  is contact depth. In this study the Oliver and Pharr method was used to calculate the initial stiffness ( $S$ ), contact depth ( $h_c$ ). The specimens were glued on metallic sample holders [5] [6].

The indentation modulus is calculated from the Plane Strain modulus using an estimated sample Poisson's ratio:

$$E_{IT} = E^* \cdot (1 - \nu_s^2) \quad (3)$$

The deduced modulus is calculated from the following equation:

$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \beta \cdot \sqrt{A_p}(h_c)} \quad (4)$$

The Plane Strain Modulus  $E^*$  is calculated from the following equation:

$$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i}} \quad (5)$$

Where  $E_i$  is the Elastic modulus of the indenter,  $E_r$  is the Reduced modulus of the indentation contact,  $\nu_i$  is the Poisson's ratio of the indenter.

Determination of indentation hardness CIT:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100 \quad (6)$$

Where  $h_1$  is the indentation depth at time  $t_1$  of reaching the test force (which is kept constant),  $h_2$  is the indentation depth at time  $t_2$  of holding the constant test force [1] [4] [5].

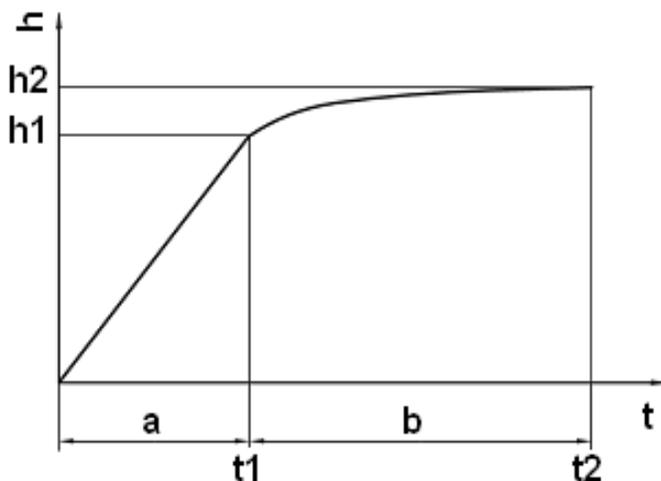


Fig. 5 Expression of indentation creep

Elastic part of the indentation work  $\eta_{IT}$ :

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} \cdot 100 \quad \text{with} \quad W_{total} = W_{elast} + W_{plast} \quad (7)$$

$$\text{Plastic part } W_{plast} / W_{total} \text{ follows as } 100\% - \eta_{IT} \quad (8)$$

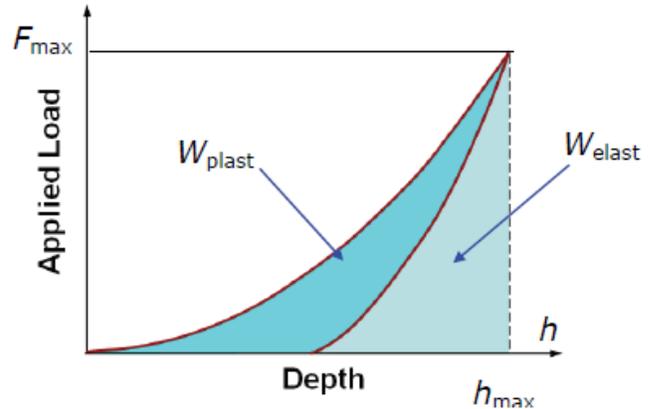


Fig. 6 Indentation work  $\eta_{IT}$

### III. RESULTS AND DISCUSSION

The figure 6 and 7 shows a very important correlation between the force and the depth of the indentation. The correlations provide very valuable information on the behaviour of tested material and the modified surface layer.

The correlation between the force and the depth of the indentation in HDPE also proved very interesting. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens. The non-irradiated material showed low hardness as well as increasing impression of the indenter in the surface layer. On the contrary, the irradiated HDPE showed considerably smaller depth of the impression of the indenter which can signify greater resistance of this layer to wear.

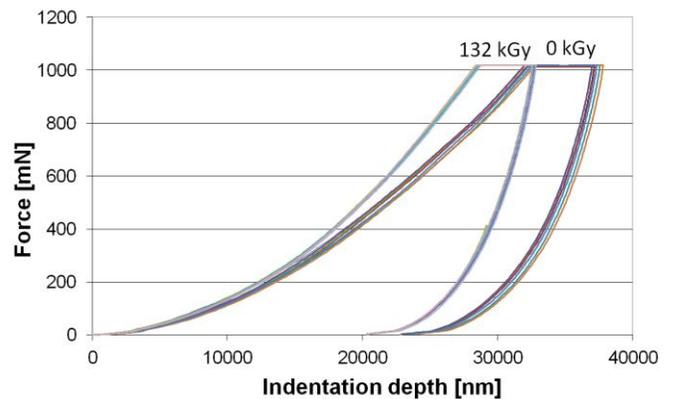


Fig. 7 Indentation depth vs. time

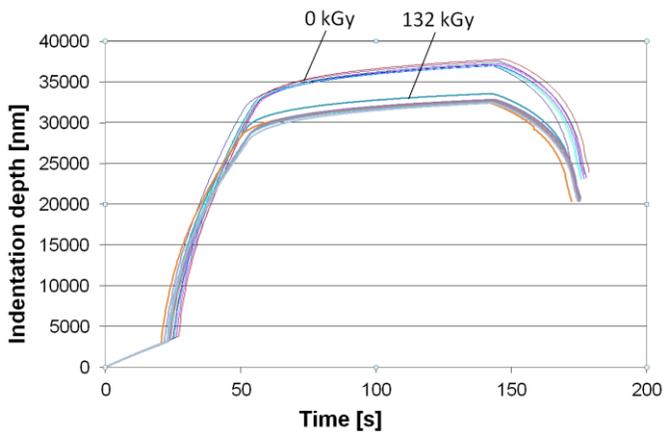


Fig. 8 Force vs. Indentation depth

A. Indentation load 0,5N

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated HDPE. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 66 kGy (by 22% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 9.

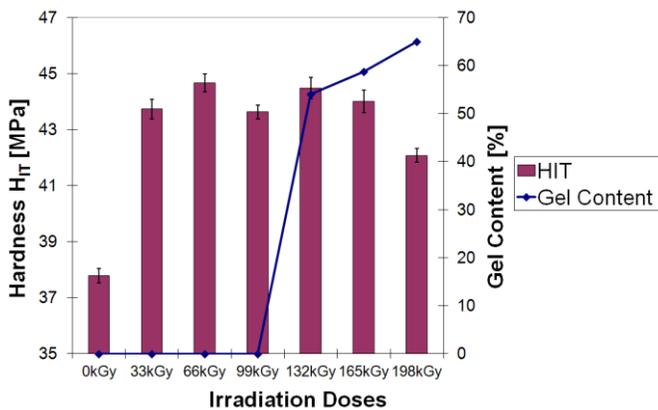


Fig. 9 Hardness HIT of HDPE vs. irradiation doses

Higher radiation dose does not influence significantly the microhardness value. An indentation hardness increase of the surface layer is caused by irradiation cross-linking of the tested specimen. A closer look at the microhardness results reveals that when the highest radiation doses are used, microhardness decreases which can be caused by radiation induced degradation of the material.

According to the results of measurements of microhardness, it was found that the highest values of indentation modulus of elasticity were achieved at the HDPE irradiated with dose of 66 kGy (by 25% higher than compared with non-irradiated HDPE). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated HDPE as is seen at Fig. 10.

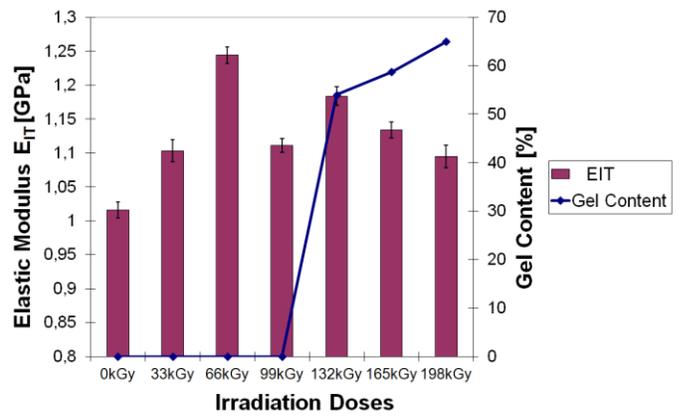


Fig. 10 Elastic modulus EIT of HDPE vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated HDPE. On the contrary, the highest values of hardness Vickers were obtained for HDPE irradiated by a dose of 66 kGy (by 17% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 11.

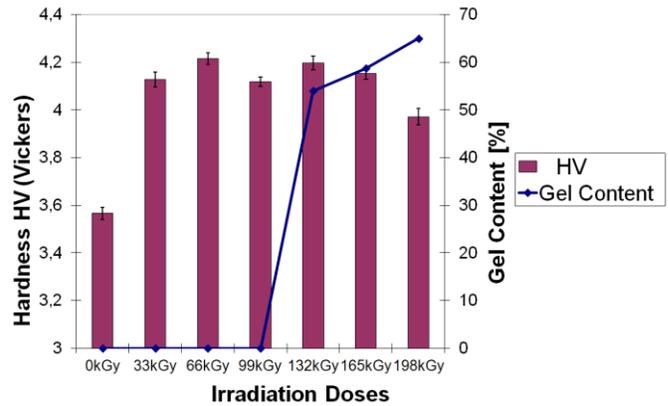


Fig. 11 Hardness Vickers of HDPE vs. irradiation doses

Other important material parameters obtained during the microhardness test were elastic and plastic deformation work. The elastic deformation work  $W_e$  determines the reaction of material to applied (multiaxial) load with reversible deformation. The plastic part of the deformation work  $W_{pl}$  defines toughness of the tested material (surface layer) and its resistance to plastic deformation (Fig. 12).

The greatest values of plastic and elastic deformation work were obtained for non-irradiated HDPE. The lowest values of both elastic and plastic deformation work were obtained for HDPE irradiated with dose of 66 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 12.

Next to plastic and elastic deformation work, the coefficient of back deformation  $\eta_{IT}$  is especially important for the assessment of the structure of the irradiated HDPE. The highest values were measured at irradiation doses of 165 kGy of HDPE. The smallest values were found at irradiation doses of 66 kGy.

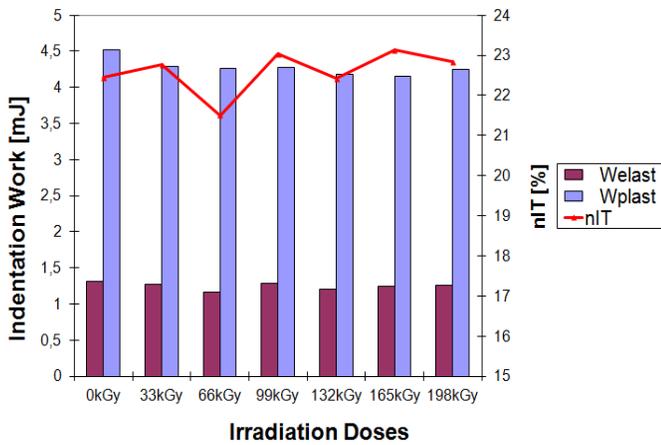


Fig. 12 Elastic and plastic deformation work of HDPE vs. irradiation dose

**B. Indentation load IN**

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated HDPE. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 132 kGy (by 16% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 13.

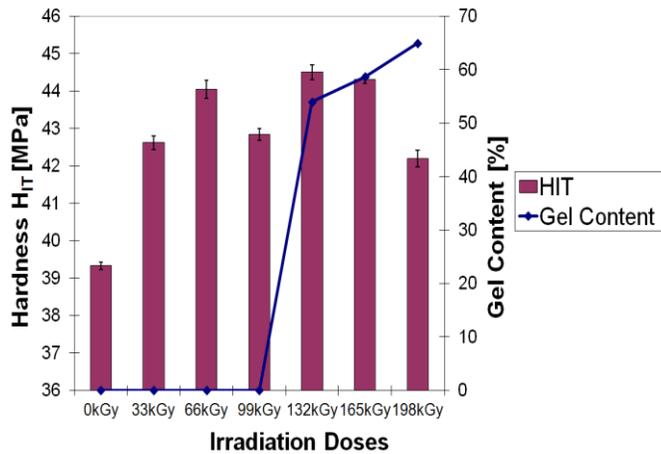


Fig. 13 Hardness HIT of HDPE vs. irradiation doses

Higher radiation dose does not influence significantly the microhardness value. An indentation hardness increase of the surface layer is caused by irradiation cross-linking of the tested specimen. A closer look at the microhardness results reveals that when the highest radiation doses are used, microhardness decreases which can be caused by radiation induced degradation of the material.

According to the results of measurements of microhardness, it was found that the highest values of indentation modulus of elasticity were achieved at the HDPE irradiated with dose of 66 kGy (by 20% higher than compared with non-irradiated HDPE). On the contrary, the lowest values

of the indentation modulus of elasticity were found for non-irradiated HDPE as is seen at Fig. 14.

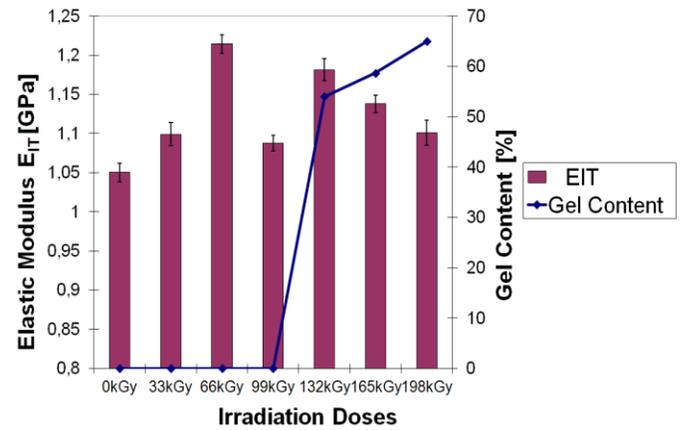


Fig. 14 Elastic modulus EIT of HDPE vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated HDPE. On the contrary, the highest values of hardness Vickers were obtained for HDPE irradiated by a dose of 132 kGy (by 14% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 15.

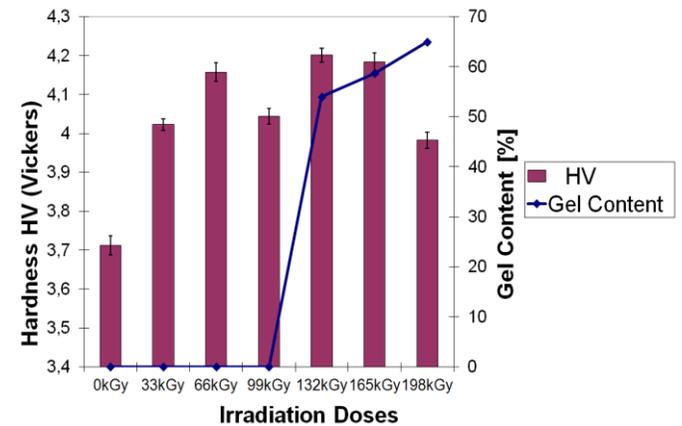


Fig. 15 Hardness Vickers of HDPE vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PP. The lowest values of both elastic and plastic deformation work were obtained for HDPE irradiated with dose of 132 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 16.

Next to plastic and elastic deformation work, the coefficient of back deformation  $\eta_{IT}$  is especially important for the assessment of the structure of the irradiated HDPE. The highest values were measured at irradiation doses of 99 kGy of HDPE. The smallest values were found at irradiation doses of 66 kGy.

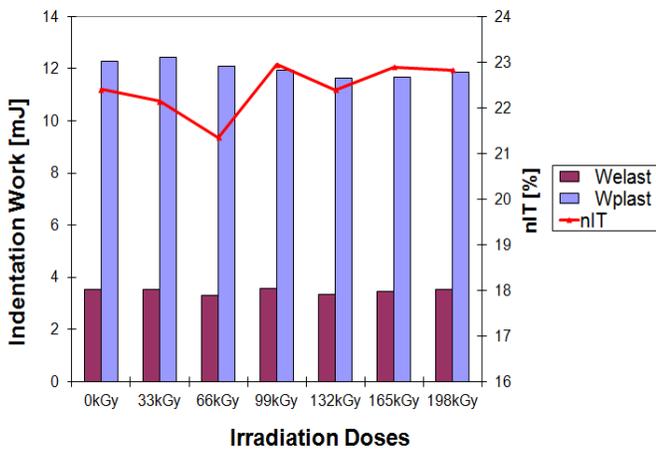


Fig. 16 Elastic and plastic deformation work of HDPE vs. irradiation dose

C. Indentation load 5N

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated HDPE. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 132 kGy (by 13% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 17.

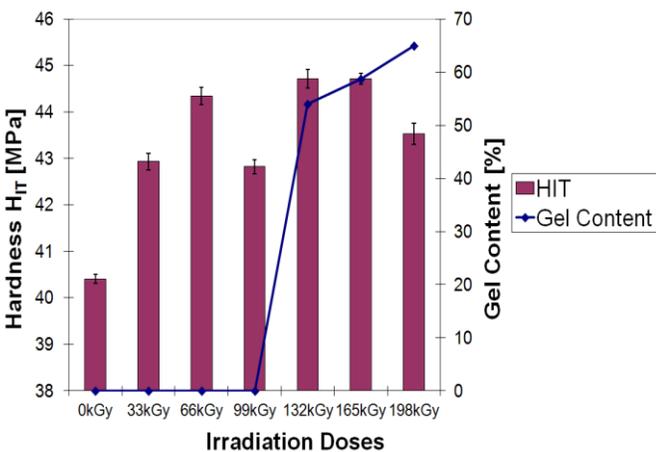


Fig. 17 Hardness HIT of HDPE vs. irradiation doses

Higher radiation dose does not influence significantly the microhardness value. An indentation hardness increase of the surface layer is caused by irradiation cross-linking of the tested specimen. A closer look at the microhardness results reveals that when the highest radiation doses are used, microhardness decreases which can be caused by radiation induced degradation of the material.

According to the results of measurements of microhardness, it was found that the highest values of indentation modulus of elasticity were achieved at the HDPE irradiated with dose of 66 kGy (by 14% higher than compared with non-irradiated HDPE). On the contrary, the lowest values

of the indentation modulus of elasticity were found for non-irradiated HDPE as is seen at Fig. 18.

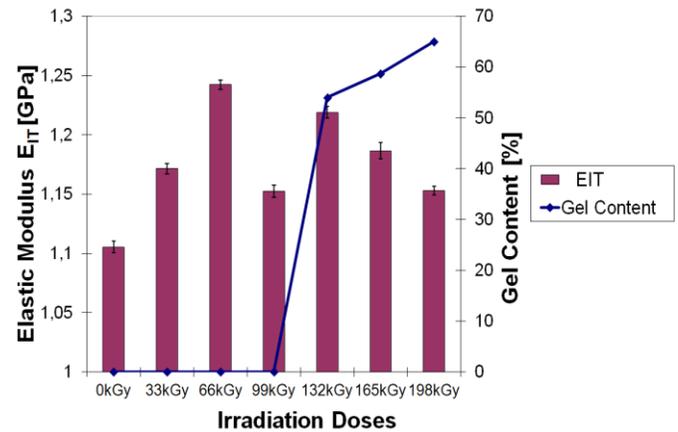


Fig. 18 Elastic modulus EIT of HDPE vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated HDPE. On the contrary, the highest values of hardness Vickers were obtained for HDPE irradiated by a dose of 132 kGy (by 11% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 19.

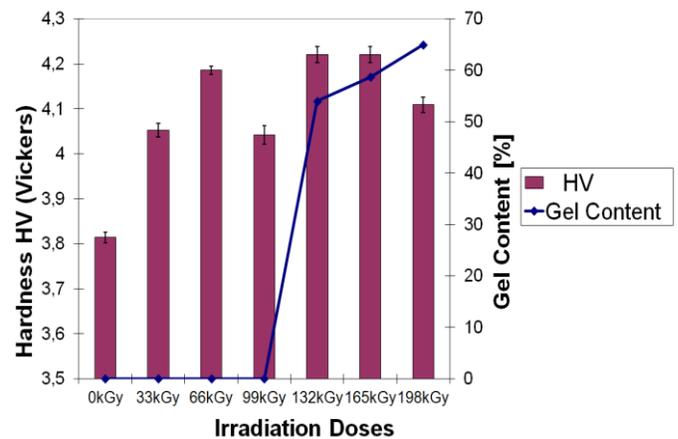


Fig. 19 Hardness Vickers of HDPE vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated HDPE. The lowest values of both elastic and plastic deformation work were obtained for HDPE irradiated with dose of 165 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 20.

Next to plastic and elastic deformation work, the coefficient of back deformation  $\eta IT$  is especially important for the assessment of the structure of the irradiated HDPE. The highest values were measured at irradiation doses of 198 kGy of HDPE. The smallest values were found at irradiation doses of 66 kGy.

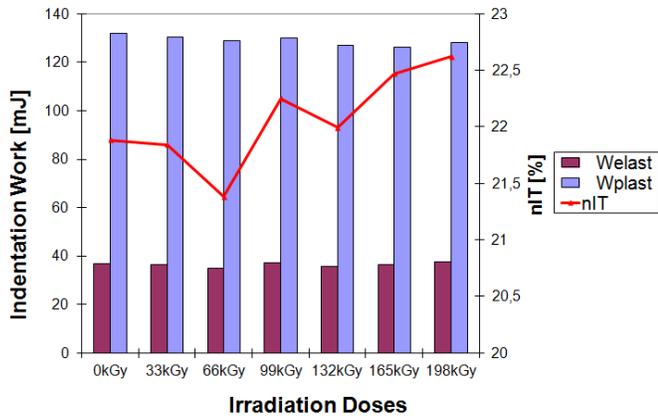


Fig. 20 Elastic and plastic deformation work of HDPE vs. irradiation dose

D. Indentation load 0,5N, 1N and 5N

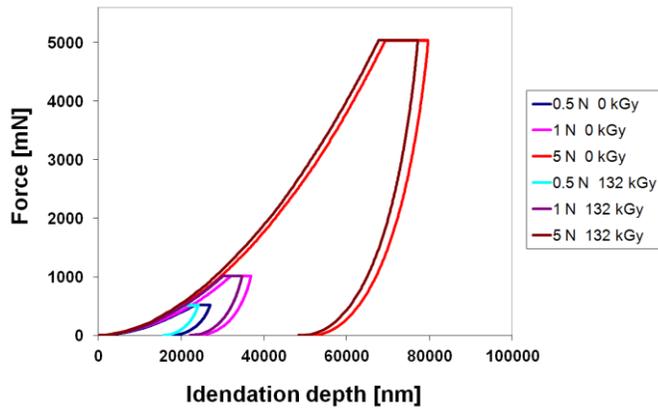


Fig. 21 Indentation depth vs. time

The figure 20 shows a very important correlation between the force and the depth of the indentation. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens. The non-irradiated material showed low hardness as well as increasing impression of the indenter in the surface layer. On the contrary, the irradiated HDPE showed considerably smaller depth of the impression of the indenter which can signify greater resistance of this layer to wear.

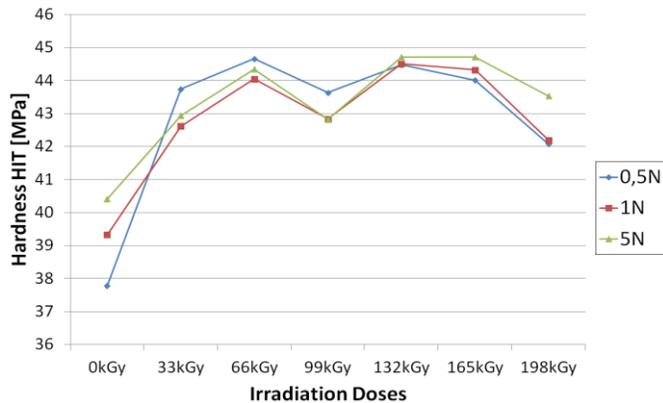


Fig. 22 Hardness HIT of HDPE vs. irradiation doses

The load applied for microhardness test was 0.5N, 1N and 5N. We observed the effect of the load on the resulting properties of the surface layer of HDPE modified by beta radiation. The measurement results show that at all loads applied the highest value of microhardness was found when the radiation dose was 132 kGy. When higher radiation doses are applied, microhardness values decline, showing constant values. At higher loads there is a slight but not significant microhardness values. They range within statistical discrepancy. The increase in microhardness values at 5N load is caused by deeper penetration of the indenter, thus reaching semicrystalline structure of HDPE tested. The increase in microhardness of the surface layer at the dose of 132 kGy compared to the non-irradiated specimen was found to be around 22%.

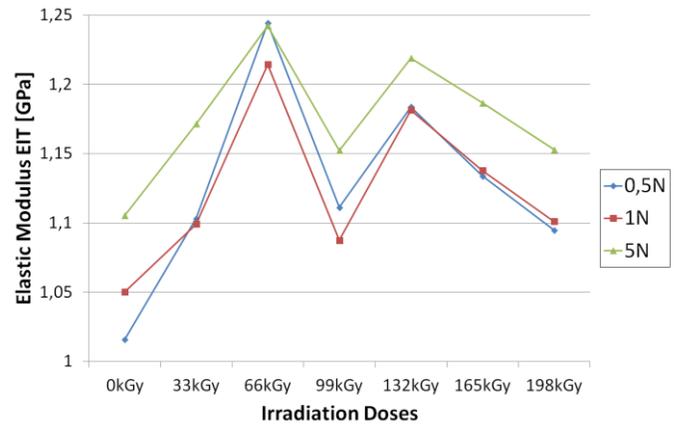


Fig. 23 Elastic modulus EIT of HDPE vs. irradiation doses

When observing the changes of stiffness of the surface layer measured by microhardness test it was proved that the maximum value of stiffness was found at radiation dose of 132 kGy, when applying all three loads (0.5N, 1N, 5N). The non-irradiated specimen showed the lowest value. At higher radiation dose, increase in the stiffness of the surface layer is not uniform. In general it can be said that stiffness of the surface layer increased by 25% in the tested specimen (132 kGy) compared to the non-irradiated specimen.

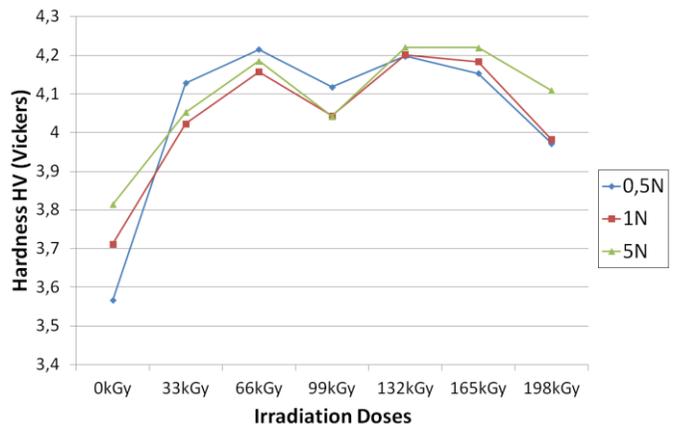


Fig. 24 Hardness Vickers of HDPE vs. irradiation doses

The results of elastic and deformation work showed that the highest values at microhardness test were found for non-irradiated specimens. The specimens subjected to beta radiation showed lower values of both elastic and plastic deformation work. The decrease in values of deformation work needed to deform the tested materials indicates changes of structure caused by radiation of the tested HDPE. The greatest changes between irradiated and non-irradiated specimen were found at 5N load. The increased radiation dose caused a slight drop of values of deformation work. This corresponded with the reverse relaxation coefficient  $\eta IT$ , which showed higher values for irradiated specimens and the lowest value for non-irradiated specimens.

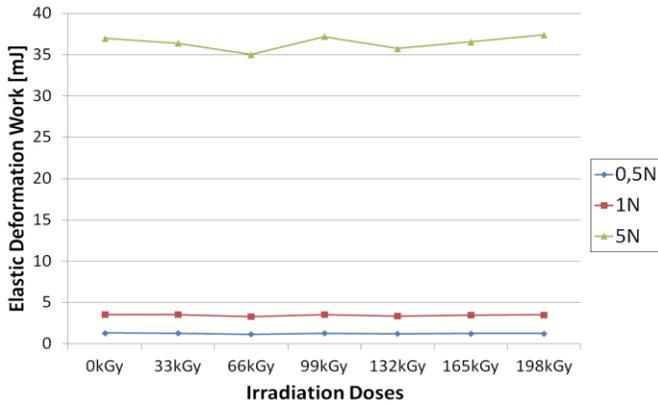


Fig. 25 Elastic deformation work of HDPE vs. irradiation dose

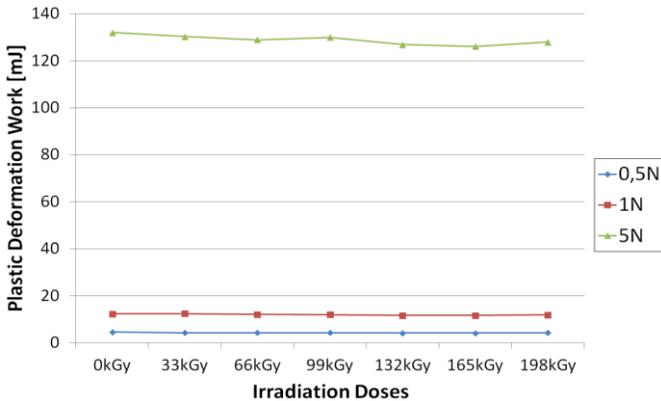


Fig. 26 Plastic deformation work of HDPE vs. irradiation dose

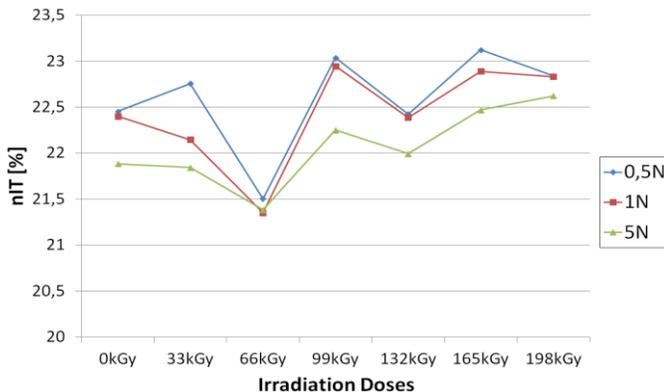


Fig. 27 Elastic part deformation work of HDPE vs. irradiation dose

E. Creep behaviour

From Figure 27, it is obvious that irradiation has a positive effect on the creep behaviour of the HDPE tested. The highest difference in indentation creep was found for an irradiation dosage of 99 kGy.

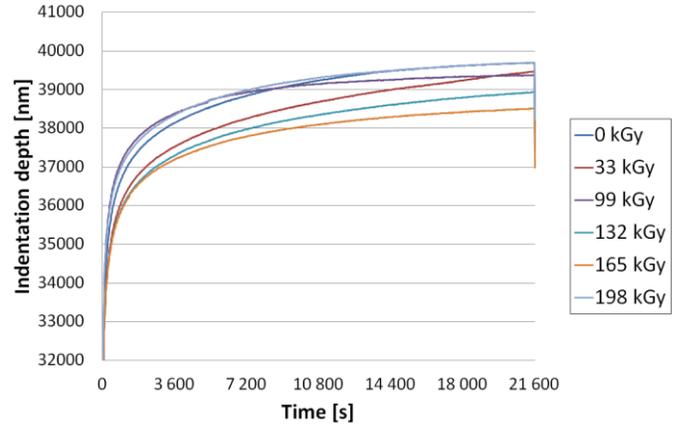


Fig. 28 Creep of HDPE

Very important values were found for indentation creep. The lowest value of creep was measured at radiation dose of 99 kGy. The highest creep value measured at radiation dose of 33 kGy for HDPE. Decrease in creep values was 25% for irradiated HDPE compared to the non-irradiated one as is seen at Fig. 29.

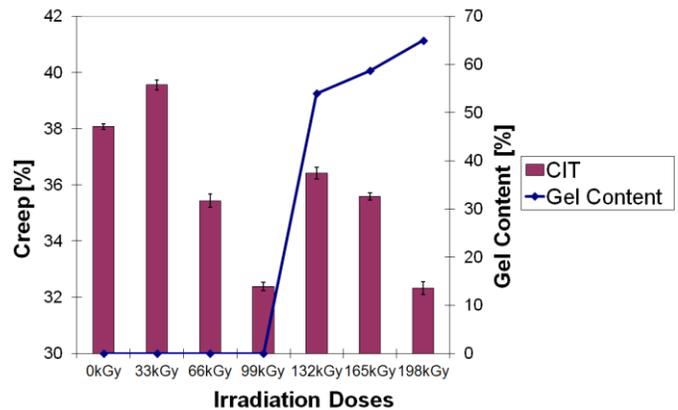


Fig. 29 Creep of HDPE vs. irradiation doses

IV. CONCLUSION

The article is the assessment of mechanical properties (microhardness) of the surface layer of modified HDPE. The surface layer of the polymer material such as HDPE is modified by  $\beta$  – radiation with doses of 33, 66, 99, 132, 165 and 199 kGy.

The properties of surface layer of HDPE modified by beta radiation improved significantly. The microhardness values increased by about 22%. Stiffness of surface layer increased significantly by 25% as a result of radiation. The creep values decreased by 25% on average for irradiated HDPE. Changes

of behavior in the surface layer were confirmed by final values of elastic and plastic deformation work whose values decreased in correlation with the increasing radiation dose. Also different depths of indentation in the surface layer of tested specimen were significantly different. The highest values of micromechanical properties were reached at radiation dose of 132 kGy. With higher radiation doses, the resulting values of micromechanical properties decreased and then showed constant values.

The results of micromechanical properties of surface layer of modified HDPE show that it can be used in more difficult applications in some industrial fields, in particular where there are high requirements for strength, stiffness and hardness of surface layer which appears to be the most suitable area of application.

#### ACKNOWLEDGMENT

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