Effect of Beta Low Irradiation Doses on the Nano-hardness of PBT

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

Abstract**—** This article deals with the influence of different doses of β - radiation on nano-mechanical properties of PBT. The PBT modification was carried out with the aid of β - radiation at different radiation intensities and the resulting properties were measured with the aid of nano-indentation test by the DSI (Depth Sensing Indentation) method. The aim of the article is to find out the influence of the radiation on the nano-hardness of the modified PBT.

Keywords**—**PBT, nano-indentation, hardness, crosslinking, β – radiation, Depth Sensing Indentation.

I. INTRODUCTION

Polybutylene terephthalate (PBT) is a semi-crystalline thermoplastic of the polyester family, which crystallizes very slowly and is therefore available in an amorphous-transparent or crystalline-opaque condition, depending on the processing method [1] [2].

PBT are characterized by their high strength and rigidity, dimensionally stable, low tendency to creep, very good frictional and wear resistance, good impact strength, very low coefficient of thermal expansion, good chemical resistance to acids, very good electrical characteristics, very low moisture absorption, good adhesion and welding ability. Furthermore, PBT, like all polyesters, has very good frictional and wearing properties. Compared to PET, PBT has a better impact strength - particularly at low temperatures [2] [3].

The irradiation cross-linking of thermoplastic materials via electron beam or cobalt 60 (gamma rays) proceeds is 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 [1] [4] [5].

The main deference between β- and γ- rays is in their different abilities of penetrating the irradiated material. γ- rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons (Fig. 1) [1] [2] [8] [9].

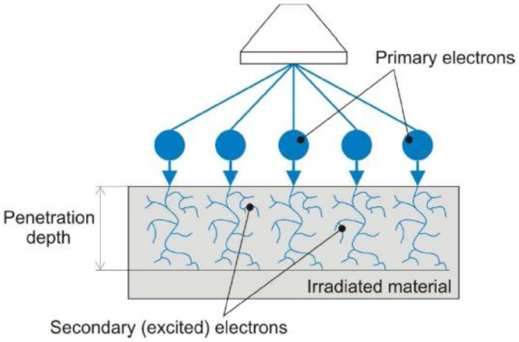

Fig. 1 Design of Electron rays

Due to electron accelerators the required dose can be applied within seconds, whereas several hours are required in the γ-radiation plant (Fig. 2) [3] [7].

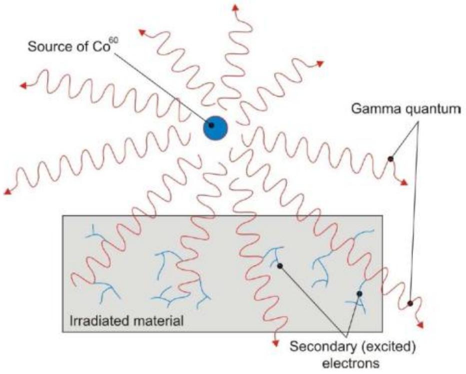

Fig. 2 Design of Gamma rays

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 [3] [8] [11].

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] [12] [14].

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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 (Tg) 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 behavior [3] [4] [17] [18].

The thermoplastics which are used for production of various types of products have very different properties. Standard polymers which are easy obtainable with favorable 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% [1] [25].

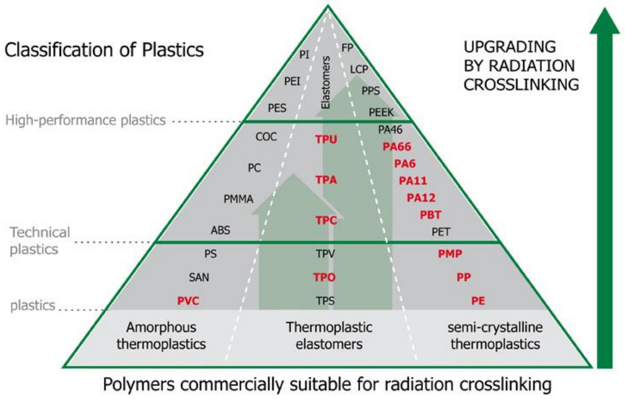

Fig. 3 Hardness H_{IT} of PA6 vs. irradiation doses

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 (Fig. 3) [1] [6].

Common PBT, when exposed to the effect of the radiation cross-linking, degrades and its mechanical properties deteriorate. Using cross-linking agent TAIC (triallyl isocyanurate) produces a cross-linking reaction inside the PBT structure. The utility properties of PBT improve when the noncrystalline part of PBT is cross-linked [4] [9].

The present experimental work deals with the influence of beta irradiation on the nano-indentation hardness of PBT.

II. Experimental

A. Irradiation

For this experiment Polybutylene terephthalate PBT V-PTS-CREATEC-B3HZC * M800/25, PTS Plastics Technology Service, Germany (unfilled, PBT+TAIC) was used. The prepared specimens were irradiated with doses of 0, 33, 66, and 99 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany.

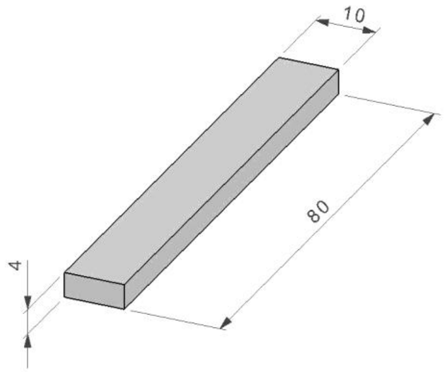

Fig. 4 Dimension of sample

B. Injection molding

The samples were made using the injection molding technology on the injection moulding machine Arburg Allrounder 420C (Fig. 4). Processing temperature 240–260 °C, mold temperature 75 °C, injection pressure 80 MPa, injection rate 65 mm/s.

C. Instrumented nanohardness tests

Instrumented nano-hardness tests were done using a Nano-indentation tester (NHT), CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1 (Fig. 5). Load and unload speed were 20 mN/min, 100 mN/min and 500 mN/min. After a holding time of 90 s at maximum load 10 mN, 50 mN and 250 mN the specimens were unloaded.

Fig. 5 Nano-indentation tester

The indentation hardness H_{IT} was calculated as maximum load to the projected area of the hardness impression according to [6] [13] [14] [15]:

$$H_{IT} = \frac{F_{\max}}{A_p} \quad \text{with} \quad h_c = h_{\max} - \varepsilon \frac{F_{\max}}{S} \qquad (1)$$

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 calculate the initial stiffness (S), contact depth (h_c). The specimens were glued on metallic sample holders (Fig. 6).

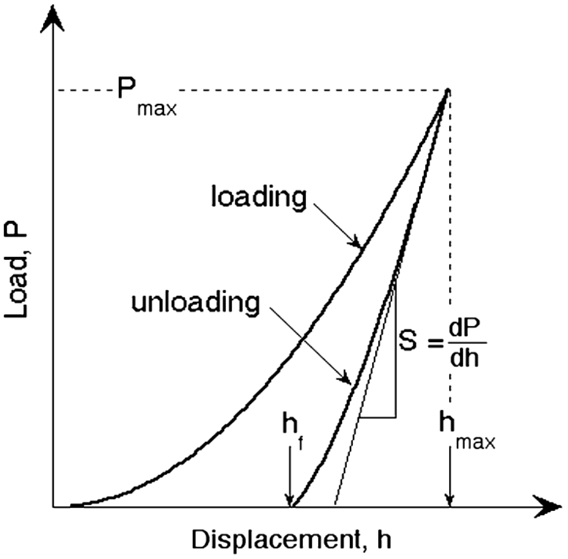

Fig. 6 Schematic illustration of indentation curve

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

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

The deduced modulus is calculated from the following equation:

$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \beta \cdot \sqrt{A_p(h_c)}} \qquad (3)$$

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

$$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i}} \qquad (4)$$

Where E_i is the Elastic modulus of the indenter, E_r is the Reduced modulus of the indentation contact, ν_i is the Poisson´s ratio of the indenter. [3] [11] [16].

Determination of indentation hardness C_{IT}:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100 \qquad (5)$$

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 (Fig. 7) [1] [7] [15].

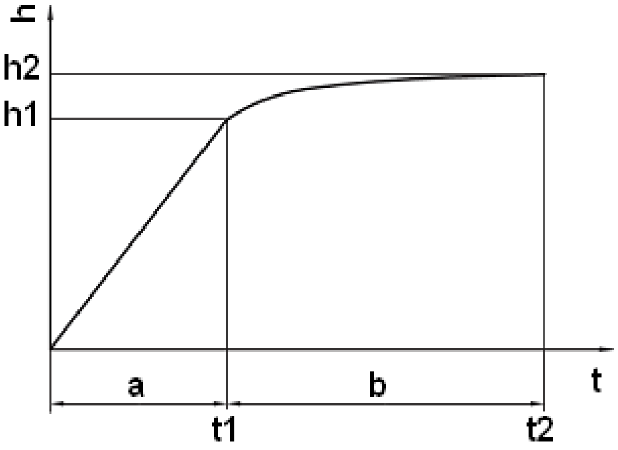

Fig. 7 Expression of indentation creep

Elastic part of the indentation work ηIT (Fig. 8):

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} \cdot 100 \quad \text{with} \quad W_{total} = W_{elast} + W_{plast} \qquad (6)$$

Plastic part W_{plast} / W_{total} follows as 100% - ηIT (7)

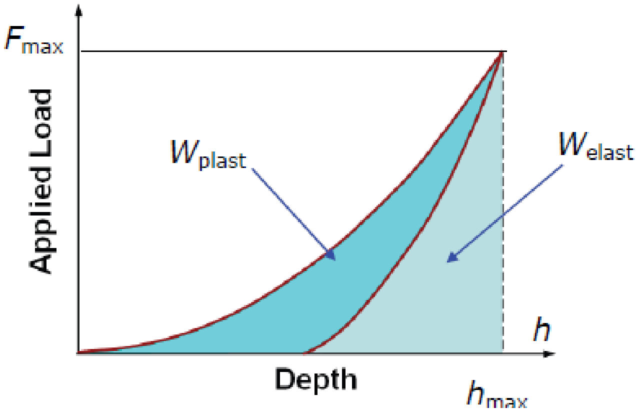

Fig. 8 Indentation work η_{IT}

III. RESULTS AND DISCUSSION

Nano-indentation characteristics determined by DSI method are depicted in Fig. 9 and 10. They characterize course of loading force in dependence on indentor penetration depth, which gives an idea about course of instantaneous values of observed nano-mechanical properties.

The correlation between the force and the depth of the nano-indentation in PBT 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 indentor in the surface layer. On the contrary, the irradiated (99 kGy) PBT showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.

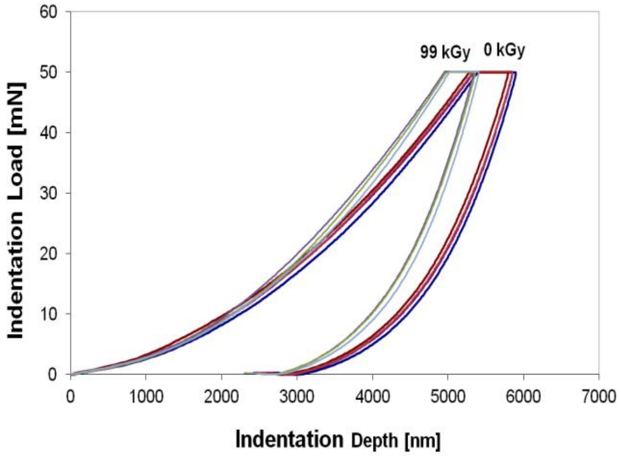

Fig. 9 Indentation load vs. Indentation depth

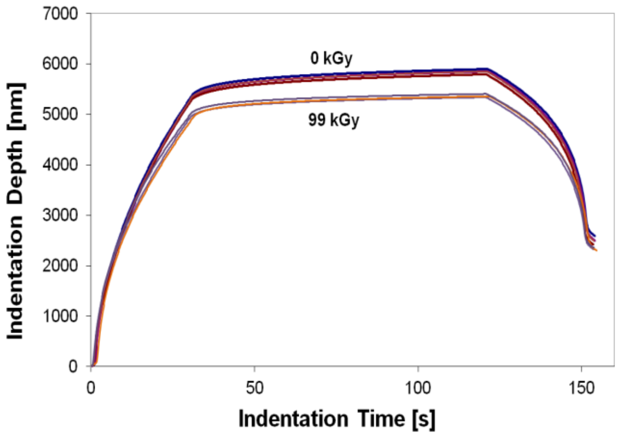

Fig. 10 Indentation depth vs. Indentation time

A. *Indentation load 10 mN*

The values measured during the nano-indentation test showed that the lowest values of indentation hardness and Vickers hardness were found for the non-irradiated PBT. On the contrary, the highest values of indentation hardness and Vickers hardness were obtained for PBT irradiated by a dose of 99 kGy (by 17% higher in comparison with the non-irradiated PBT), as can be seen at Fig. 11.

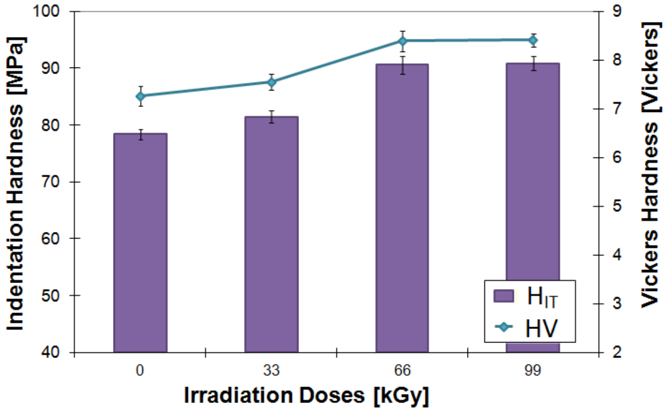

Fig. 11 Hardness H_{IT} of PBT vs. irradiation doses

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

According to the results of measurements of nano-indentation test, it was found that the highest values of indentation modulus of elasticity were achieved at the PBT irradiated with dose of 66 kGy and 99 kGy (by 14% higher than compared with non-irradiated PBT). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PBT as is seen at Fig. 12.

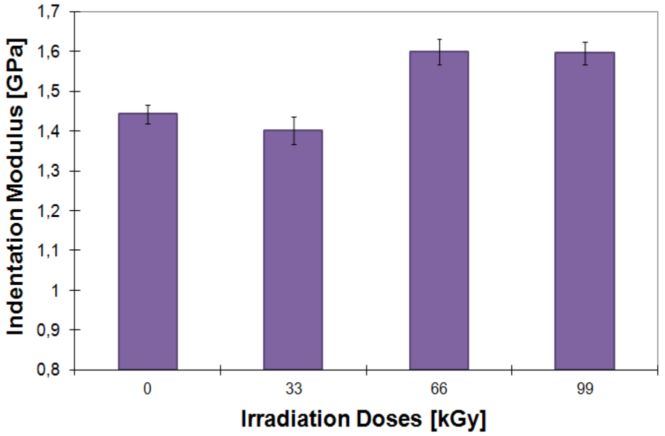

Fig. 12 Elastic modulus E_{IT} of PBT vs. irradiation doses

Other important material parameters obtained during the nano-indentation test were elastic (W_{el}) and plastic deformation work (W_{pl}). The greatest values of plastic and elastic deformation work were obtained for non-irradiated PBT. The lowest values of both elastic and plastic deformation work were obtained for PBT irradiated with dose of 99 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 13.

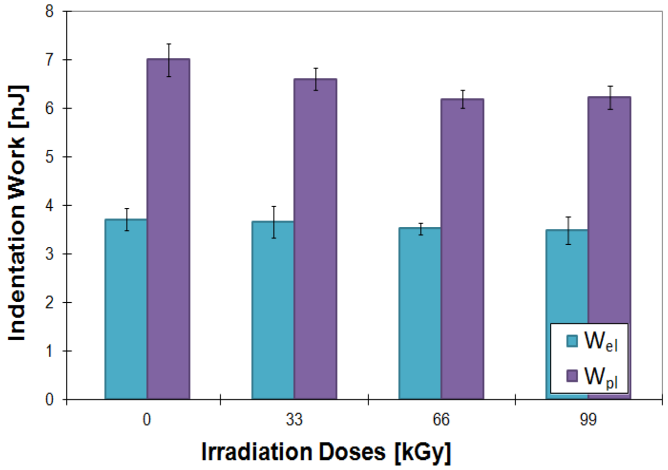

Fig. 13 Deformation work vs. irradiation dose

B. Indentation load 50 mN

The values measured during the nano-indentation test showed that the lowest values of indentation hardness and Vickers hardness were found for the non-irradiated PBT. On the contrary, the highest values of indentation hardness and Vickers hardness were obtained for PBT irradiated by a dose of 99 kGy (by 13% higher in comparison with the non-irradiated PBT), as can be seen at Fig. 14.

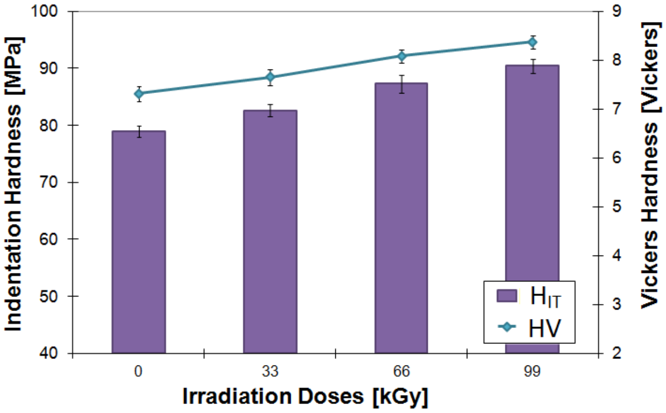

Fig. 14 Hardness H_{IT} of PBT vs. irradiation doses

According to the results of measurements of nano-indentation test, it was found that the highest values of indentation modulus of elasticity were achieved at the PBT irradiated with dose of 99 kGy (by 20% higher than compared with non-irradiated PBT). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PBT as is seen at Fig. 15.

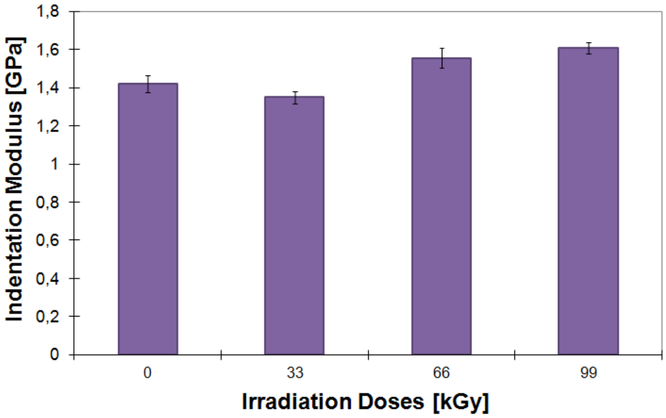

Fig. 15 Elastic modulus E_{IT} of PBT vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PBT. The lowest values of both elastic and plastic deformation work were obtained for PBT irradiated with dose of 99 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 16.

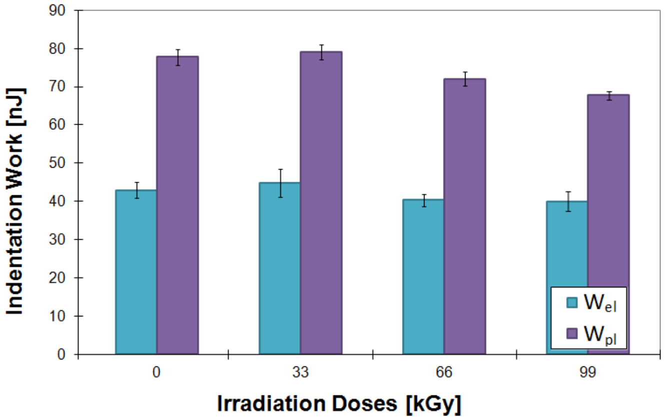

Fig. 16 Deformation work vs. irradiation dose

C. Indentation load 250 mN

The values measured during the nano-indentation test showed that the lowest values of indentation hardness and Vickers hardness were found for the non-irradiated PBT. On the contrary, the highest values of indentation hardness and Vickers hardness were obtained for PBT irradiated by a dose of 66 kGy (by 15% higher in comparison with the non-irradiated PBT), as can be seen at Fig. 17.

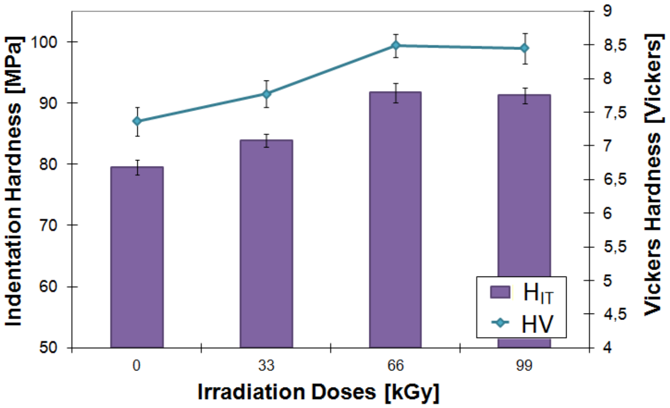

Fig. 17 Hardness H_{IT} of PBT vs. irradiation doses

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

According to the results of measurements of nano-indentation test, it was found that the highest values of indentation modulus of elasticity were achieved at the PBT irradiated with dose of 66 kGy (by 10% higher than compared with non-irradiated PBT). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PBT as is seen at Fig. 18.

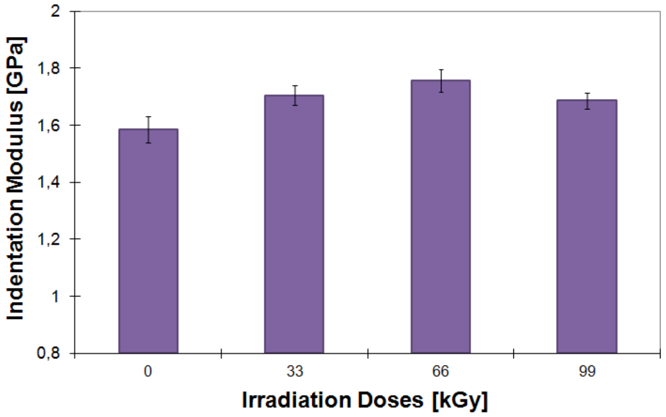

Fig. 18 Elastic modulus E_{IT} of PBT vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PBT. The lowest values of both elastic and plastic deformation work were obtained for PBT 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. 19.

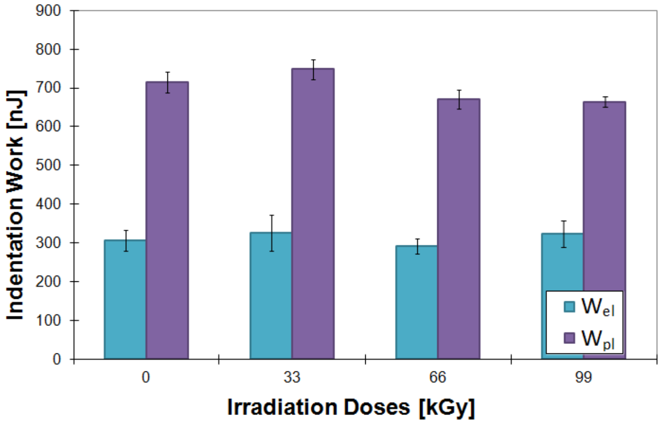

Fig. 19 Deformation work vs. irradiation dose

D. Indentation load 10 mN, 50 mN and 250 mN

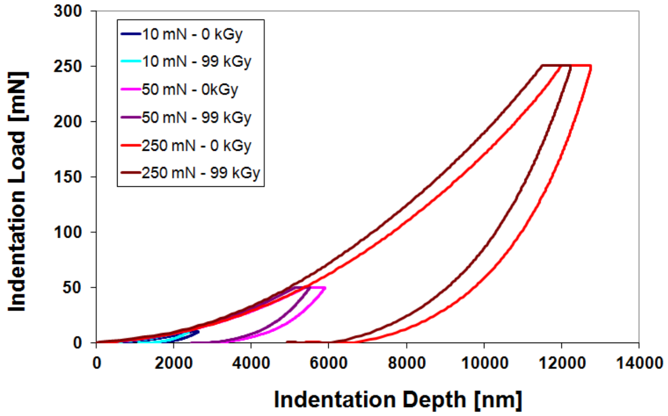

Fig. 20 Indentation load vs. Indentation depth

The figure 20 and 21 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 indentor in the surface layer. On the contrary, the irradiated PBT showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.

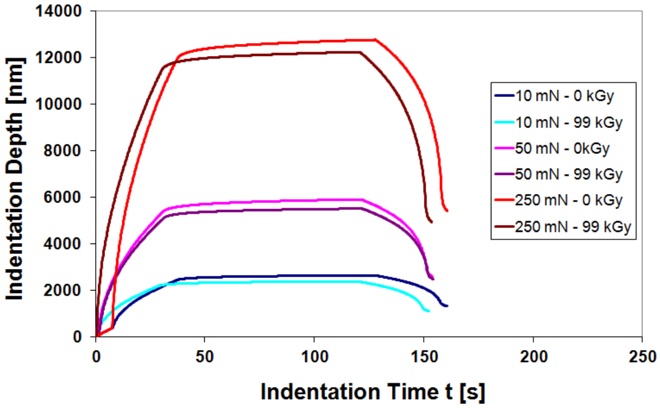

Fig. 21 Indentation depth vs. Indentation time

The load applied for nano-hardness test was 10 mN, 50 mN and 250 mN. We observed the effect of the load on the resulting properties of the surface layer of PBT modified by beta radiation. The measurement results show that at all loads applied the highest value of nano-hardness was found when the radiation dose was 66 and 99 kGy. When higher radiation doses are applied, nano-hardness values decline, showing constant values. At higher loads there is a slight but not significant nano-hardness values. They range within statistical discrepancy. The increase in nano-hardness values at 250 mN load is caused by deeper penetration of the indentor, thus reaching semicrystalline structure of PBT. The increase in nano-hardness of the surface layer at the dose of 99 kGy compared to the non-irradiated specimen was found to be around 17% (Fig. 22).

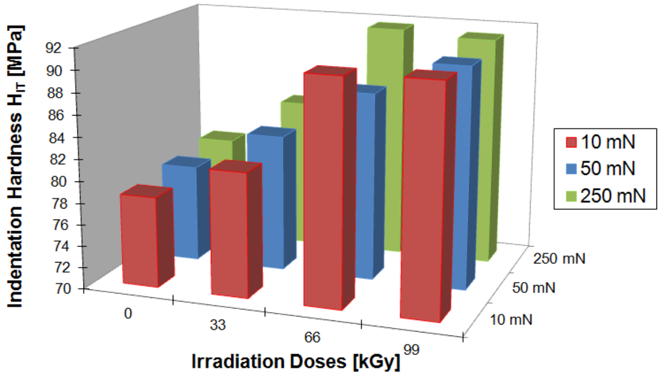

Fig. 22 Hardness H_{IT} of PBT vs. irradiation doses

When observing the changes of stiffness of the surface layer measured by nano-hardness test it was proved that the maximum value of stiffness was found at radiation dose of 66 kGy, when applying all three loads (10 mN, 50 mN, 250 mN). 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 20% in the tested specimen (99 kGy) compared to the non-irradiated specimen (Fig. 23).

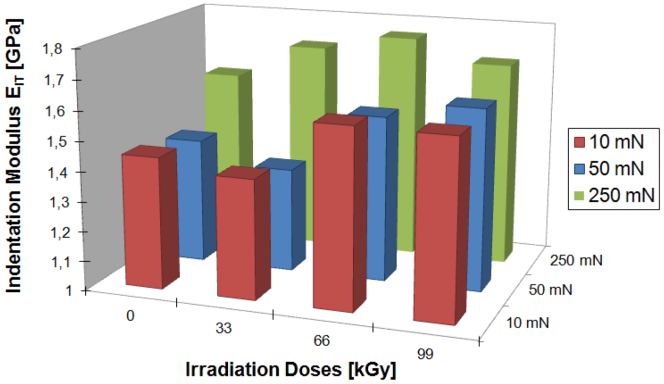

Fig. 23 Elastic modulus E_{IT} of PBT vs. irradiation doses

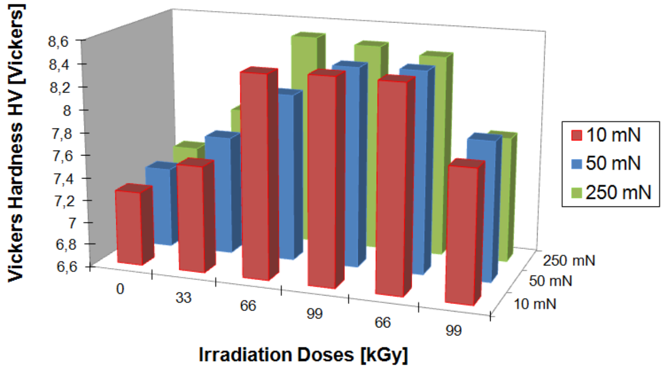

Fig. 24 Hardness Vickers of PBT vs. irradiation doses

The results of elastic and plastic deformation work showed that the highest values at nano-hardness 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 PBT. The greatest changes between irradiated and non-irradiated specimen were found at 250 mN load. The increased radiation dose caused a slight drop of values of deformation work. This corresponded with the reverse relaxation coefficient η_{IT}, which showed higher values for irradiated specimens and the lowest value for non-irradiated specimens (Fig. 25).

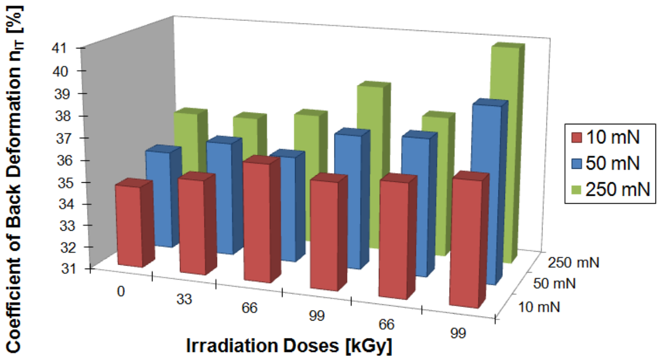

Fig. 25 Deformation work of PBT vs. irradiation dose

E. Creep behaviour

From Figure 26, it is obvious that irradiation has a positive effect on the creep behaviour of the PBT tested. The highest difference in indentation creep was found for an irradiation dosage of 99 kGy.

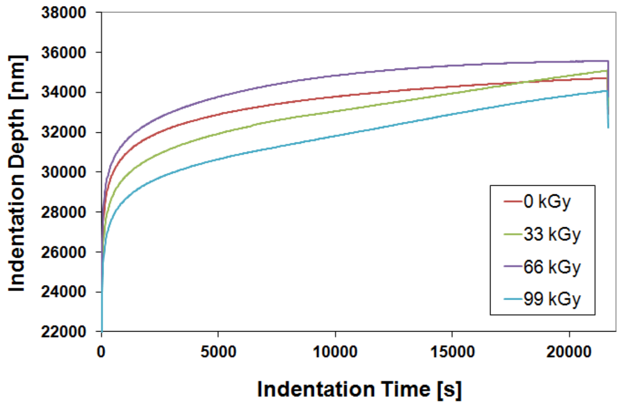

Fig. 26 Indentation creep of PBT

According to the results of measurements of nano-hardness, it was found that the lowest values of indentation creep were achieved at the PBT irradiated with dose of 99 kGy (by 25% lower than compared with non-irradiated PBT). On the contrary, the highest values of the indentation creep were found for PBT irradiated with dose of 33 kGy as is seen on Fig. 27.

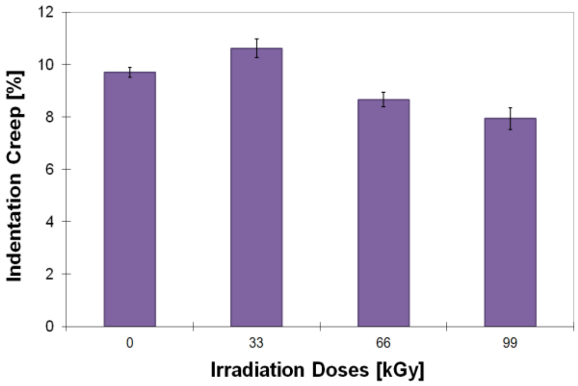

Fig. 27 Creep of PBT vs. irradiation doses

IV. CONCLUSION

The article is the assessment of mechanical properties (nano-hardness) of the surface layer of modified PBT. The surface layer of the polymer material such as PBT is modified by β – radiation with doses of 33, 66 and 99 kGy.

The properties of surface layer of PBT modified by beta radiation improved significantly. The nano-hardness values increased by about 17%. Stiffness of surface layer increased significantly by 20% as a result of radiation. The creep values decreased by 25% on average for irradiated PBT. 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 nano-mechanical properties were reached at radiation dose of 99 kGy. It also proved the fact that higher doses of radiation do not have very positive effects on the mechanical properties, on the contrary due to degradation processes the properties deteriorate.

The results of nano-mechanical properties of surface layer of modified PBT 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.

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