Microhardness of PA6 Influenced by Beta Low Irradiation Doses

M. Ovsik, D. Manas, M. Manas, M. Stanek, S. Sanda, K. Kyas and M. Reznicek

Abstract**—**The experimental study deals with the effect of modification of the surface layer by irradiation cross-linking on the mechanical properties of the PA6 tested using the instrumented microhardness test. The surface layer of PA6 specimen made by injection technology was modified by irradiation cross-linking using beta irradiation, which significantly influences mechanical properties of the surface layer. Compared to the heat and chemical-heat treatment of metal materials (e.g. hardening, nitridation, case hardening), cross-linking in polymers affects the surfaces in micro layers. These mechanical changes of the surface layer are observed in the instrumented microhardness test. Our research confirms the comparable properties of surface layer of irradiated PA6 with highly efficient polymers. The subject of this research is the influence of irradiation dosage on the changes of mechanical properties of PA6.

Keywords**—C**rosslinking, irradiation, microhardness, polyamide 6.

I. Introduction

Polyamides (PA) are semi-crystalline polymers. A distinction is made between two types. Polyamides made of one basic material (PA 6) and polyamides, which are made of 2 basic materials (PA 66). Polyamides have very good mechanical properties, are particularly tough and have excellent sliding and wear characteristics.

Polyamide 6 (PA 6) is the most common extruded polyamide and offers a balanced combination of all typical characteristics of this group of materials. Damping properties and impact strength of the material deserve to be emphasized as much as high tenacity even at low temperatures. Good abrasion resistance, particularly against sliding friction partners with rough surfaces, rounds off the overall picture.

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] [2].

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.

Due to electron accelerators the required dose can be applied within seconds, whereas several hours are required in the γ-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.

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] [9].

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 behaviour [2] [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%.

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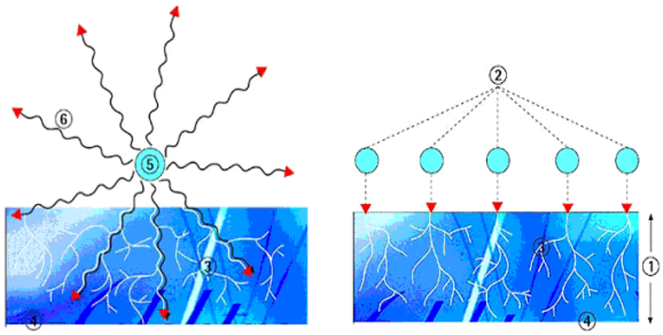

a) b)

Fig. 1 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%.

Common PA6, 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 PA6 structure. The utility properties of PA6 improve when the noncrystalline part of PA6 is cross-linked.

The present experimental work deals with the influence of beta irradiation on the microhardness of PA6.

II. Experimental

A. Irradiation

For this experiment polyamide PA6 V-PTS –Creamid-B3H2, PTS-Plastics Technologie Service, Germany (unfilled, PA6+TAIC) was used. The material already contained the special cross-linking agent TAIC - triallyl isocyanurate (6 volume %), which should enable subsequent cross-linking by ionizing β – radiation. The prepared specimens were irradiated with doses of 0,15, 30, 45, 66 and 99 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 220–280 °C, mold temperature 70 °C, injection pressure 65 MPa, injection rate 45 mm/s.

C. Micro-hardness according to Vickers

Test of hardness according to Vickers is prescribed by European standard ČSN 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°) 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°).

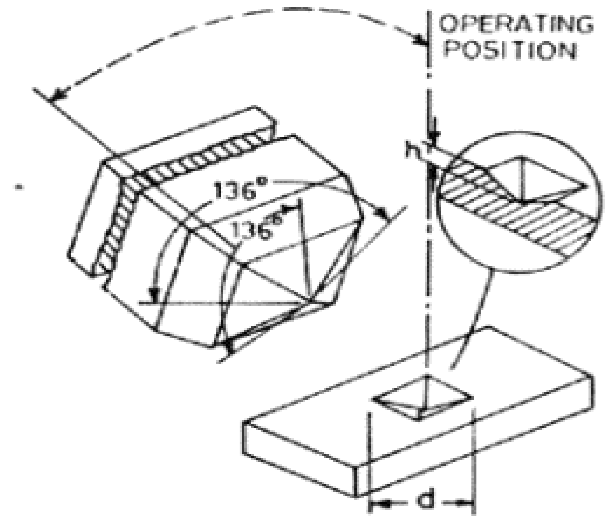

Fig. 2 The basic principle of hardness testing according to Vickers

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.

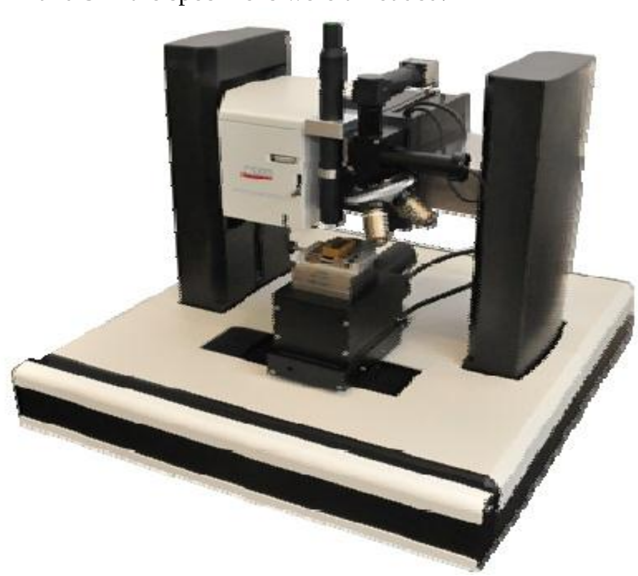

Fig. 3 Micro-combi tester

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

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

where hmax is the indentation depth at Fmax, hc is contact depth. In this study the Oliver and Pharr method was used calculate the initial stiffness (S), contact depth (hc). 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:

$$E_{IT} = E^* \cdot (1 - v_s^{\,2}) \tag{3}$$

The deduced modulus is calculated from the following equation:

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

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

$$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}} \tag{5}$$

Where Ei is the Elastic modulus of the indenter, Er is the Reduced modulus of the indentation contact, vi is the Poisson´s ratio of the indenter.

Determination of indentation hardness CIT:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100 \tag{6}$$

Where h1 is the indentation depth at time t1 of reaching the test force (which is kept constant), h2 is the indentation depth at time t2 of holding the constant test force [1] [4] [5].

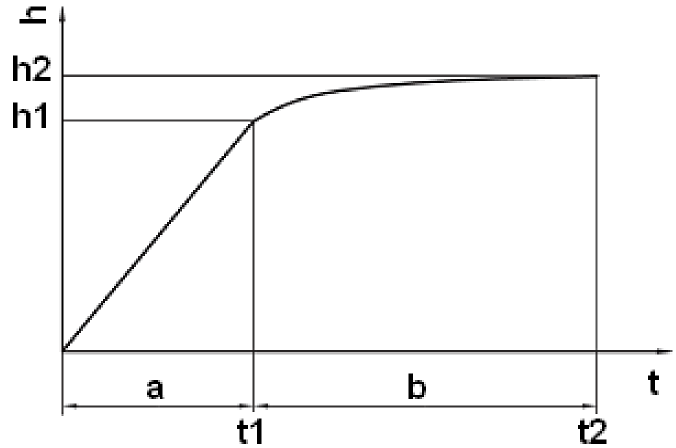

Fig. 4 Expression of indentation creep

Elastic part of the indentation work ηIT:

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

Plastic part W_{plast} / W_{total} follows as 100% - ηIT (8)

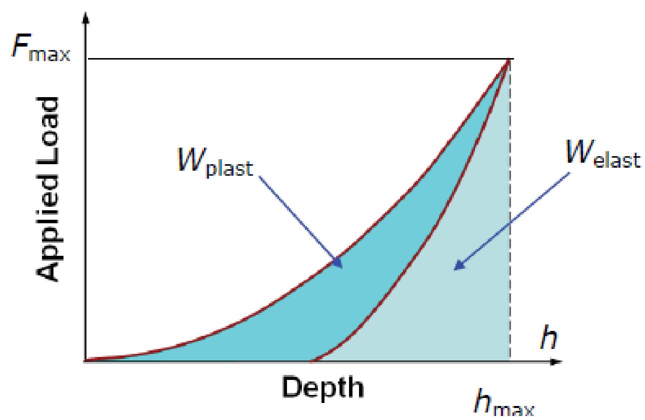

Fig. 5 Indentation work η_{IT}

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 PA6 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 PA6 showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.

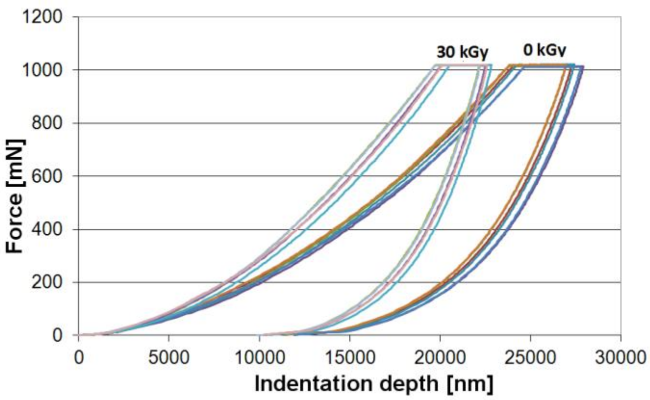

Fig. 6 Indentation depth vs. time

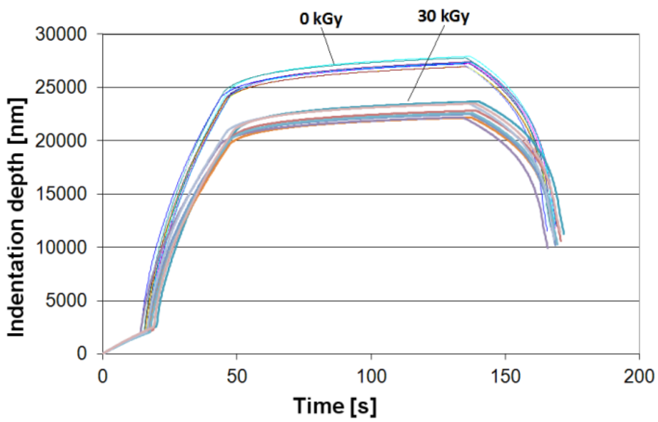

Fig. 7 Force vs. Indentation depth

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 PA6. On the contrary, the highest values of indentation hardness were obtained for PA6 irradiated by a dose of 66 kGy (by 40% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 8.

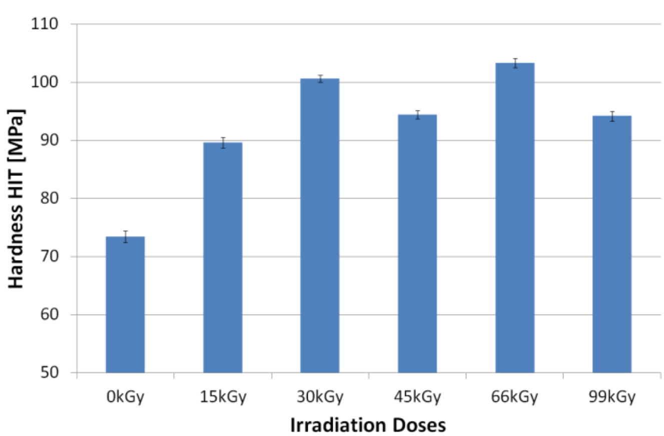

Fig. 8 Hardness HIT of PA6 vs. irradiation doses

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 indusced 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 PA6 irradiated with dose of 66 kGy (by 46% higher than compared with non-irradiated PA6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6 as is seen at Fig. 9.

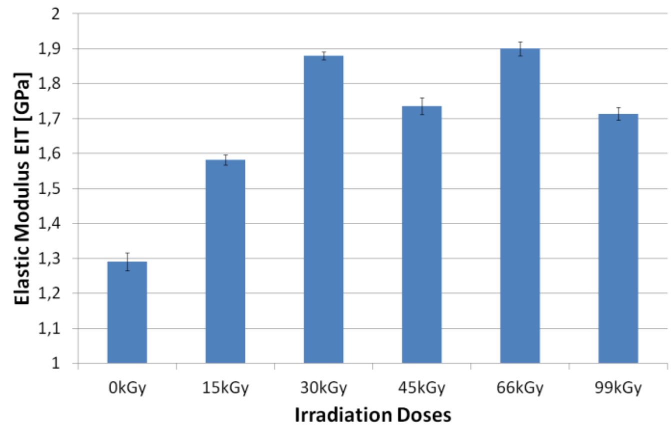

Fig. 9 Elastic modulus EIT of PA6 vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated PA6. On the contrary, the highest values of hardness Vickers were obtained for PA6 irradiated by a dose of 66 kGy (by 43% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 10.

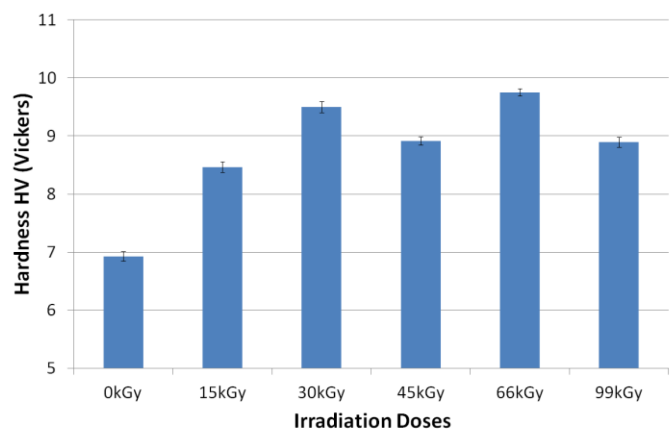

Fig. 10 Hardness Vickers of PA6 vs. irradiation doses

Other important material parameters obtained during the microhardness test were elastic and plastic deformation work. The elastic deformation work We determines the reaction of material to applied (multiaxial) load with reversible deformation. The plastic part of the deformation work Wpl defines toughness of the tested material (surface layer) and its resistance to plastic deformation (Fig. 11).

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PA6. The lowest values of both elastic and plastic deformation work were obtained for PA6 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. 11.

Next to plastic and elastic deformation work, the coefficient of back deformation ηIT is especially important for the assessment of the structure of the irradiated PA6. The highest values were measured at irradiation doses of 15 kGy of PA6. The smallest values were found at irradiation doses of 30 kGy.

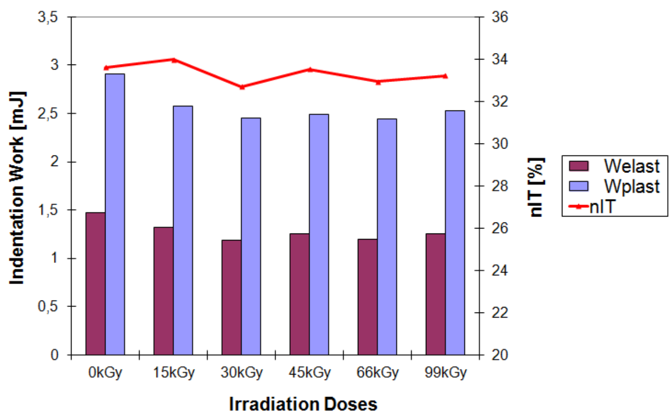

Fig. 11 Elastic and plastic deformation work of PA6 vs. irradiation dose

B. *Indentation load 1N*

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated PA6. On the contrary, the highest values of indentation hardness were obtained for PA6 irradiated by a dose of 30 kGy (by 38% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 12.

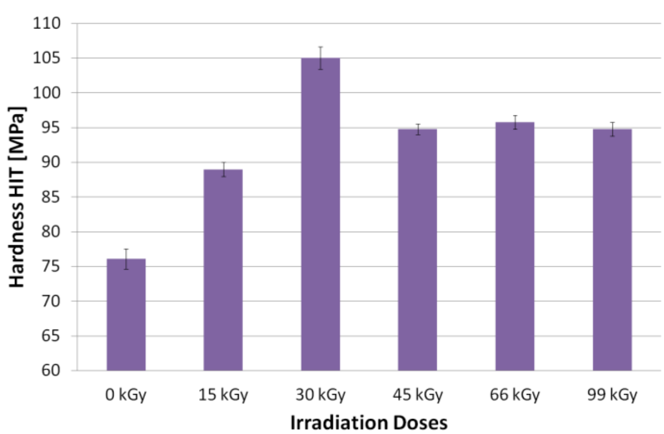

Fig. 12 Hardness HIT of PA6 vs. irradiation doses

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 indusced 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 PA6 irradiated with dose of 30 kGy (by 50% higher than compared with non-irradiated PA6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6 as is seen at Fig. 13.

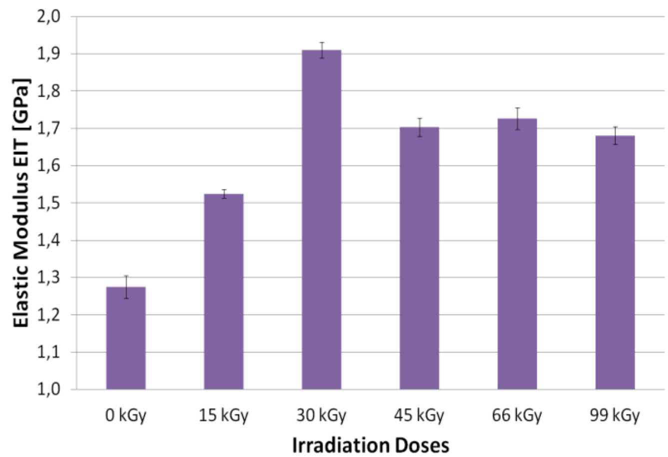

Fig. 13 Elastic modulus EIT of PA6 vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated PA6. On the contrary, the highest values of hardness Vickers were obtained for PA6 irradiated by a dose of 30 kGy (by 43% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 14.

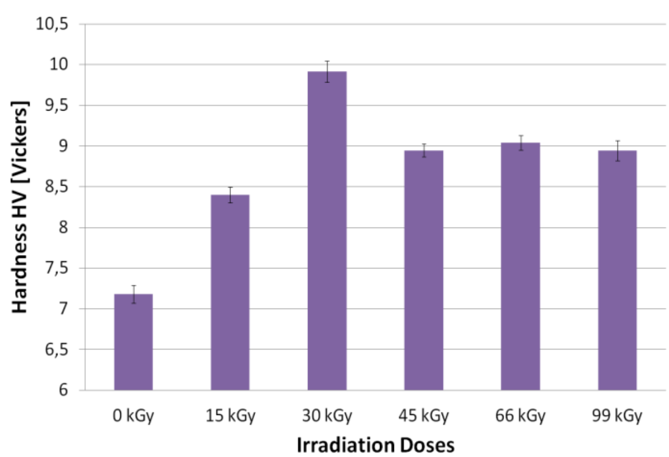

Fig. 14 Hardness Vickers of PA6 vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PA6. The lowest values of both elastic and plastic deformation work were obtained for PA6 irradiated with dose of 30 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 15.

Next to plastic and elastic deformation work, the coefficient of back deformation ηIT is especially important for the assessment of the structure of the irradiated PA6. The highest values were measured at non-irradiated PA6. The smallest values were found at irradiation doses of 30 kGy.

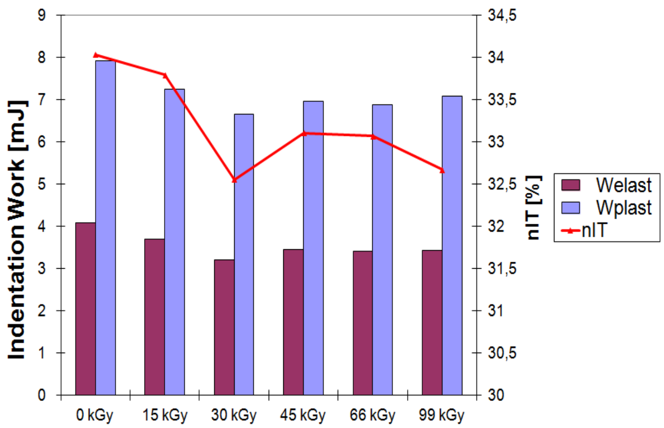

Fig. 15 Elastic and plastic deformation work of PA6 vs. irradiation dose

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 PA6. On the contrary, the highest values of indentation hardness were obtained for PA6 irradiated by a dose of 30 kGy (by 37% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 16.

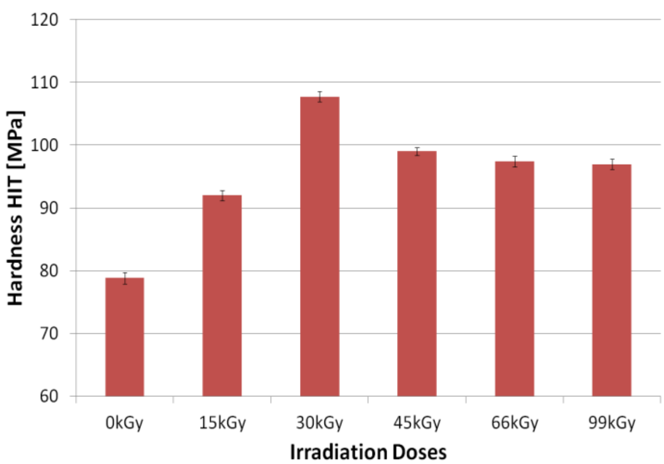

Fig. 16 Hardness HIT of PA6 vs. irradiation doses

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 indusced 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 PA6 irradiated with dose of 30 kGy (by 46% higher than compared with non-irradiated PA6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6 as is seen at Fig. 17.

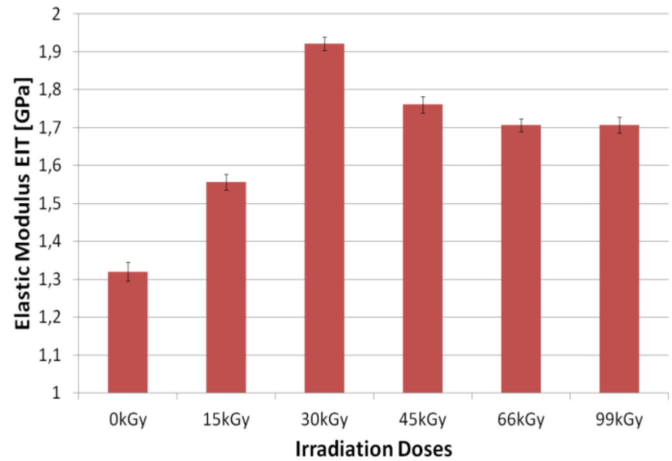

Fig. 17 Elastic modulus EIT of PA6 vs. irradiation doses

The lowest values of hardness Vickers were found for the non-irradiated PA6. On the contrary, the highest values of hardness Vickers were obtained for PA6 irradiated by a dose of 30 kGy (by 36% higher in comparison with the non-irradiated PA6), as can be seen at Fig. 18.

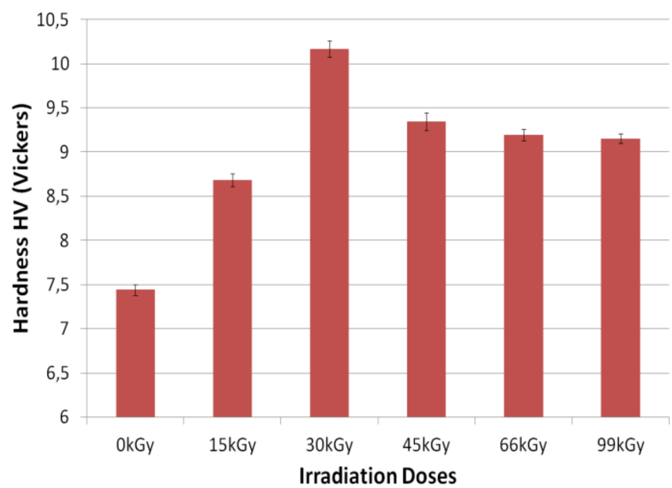

Fig. 18 Hardness Vickers of PA6 vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for non-irradiated PA6. The lowest values of both elastic and plastic deformation work were obtained for PA6 irradiated with dose of 30 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 19.

Next to plastic and elastic deformation work, the coefficient of back deformation ηIT is especially important for the assessment of the structure of the irradiated PA6. The highest values were measured at non-irradiated PA6. The smallest values were found at irradiation doses of 99 kGy.

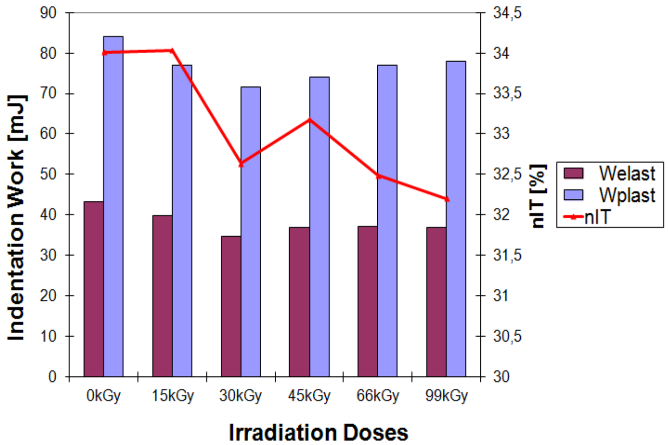

Fig. 19 Elastic and plastic deformation work of PA6 vs. irradiation dose

D. Indentation Load 0.5N, 1N and 5N

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 indentor in the surface layer. On the contrary, the irradiated PA6 showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.

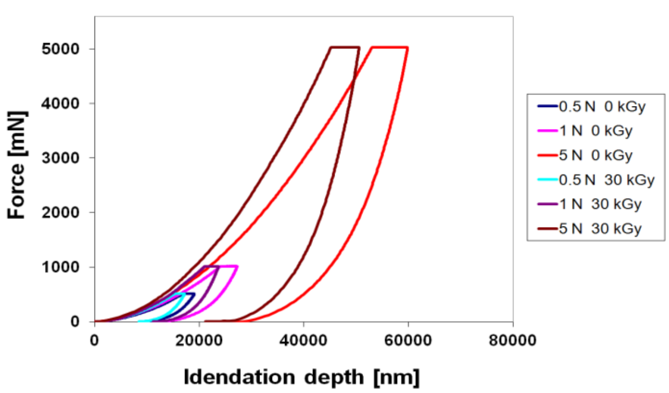

Fig. 20 Indentation depth vs. time

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 polyamide 6 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 30 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 indentor, thus reaching semicrystalline structure of polyamide 6 tested. The increase in microhardness of the surface layer at the dose of 30 kGy compared to the non-irradiated specimen was found to be around 41%.

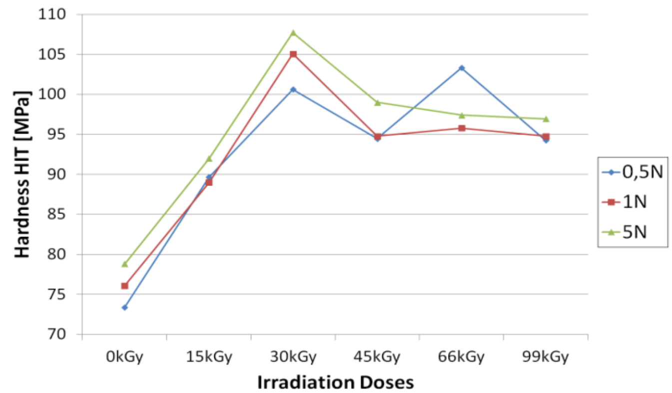

Fig. 21 Hardness HIT of PA6 vs. irradiation doses

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 30 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 50% in the tested specimen (30 kGy) compared to the non-irradiated specimen.

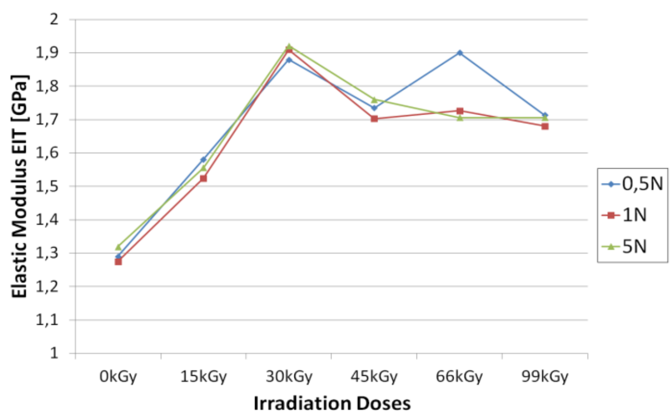

Fig. 22 Elastic modulus EIT of PA6 vs. irradiation doses

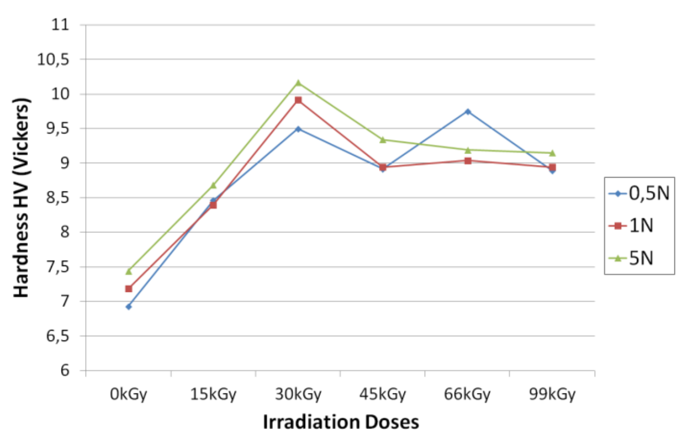

Fig. 23 Hardness Vickers of PA6 vs. irradiation doses

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 polyamide 6. 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 ηIT, which showed higher values for irradiated specimens and the lowest value for non-irradiated specimens.

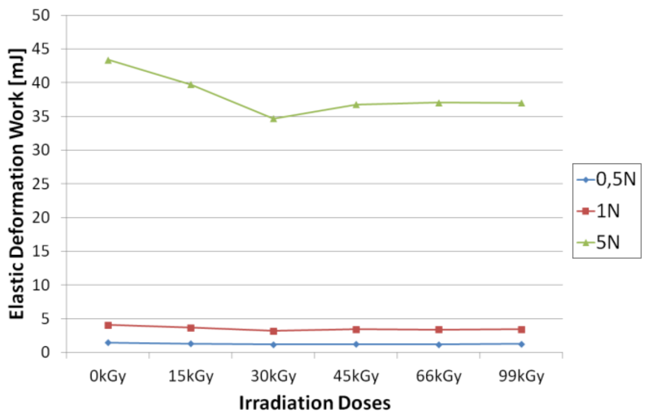

Fig. 24 Elastic deformation work of PA6 vs. irradiation dose

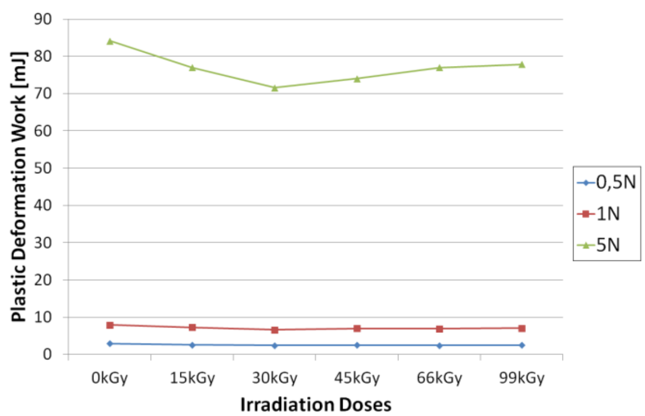

Fig. 25 Plastic deformation work of PA6 vs. irradiation dose

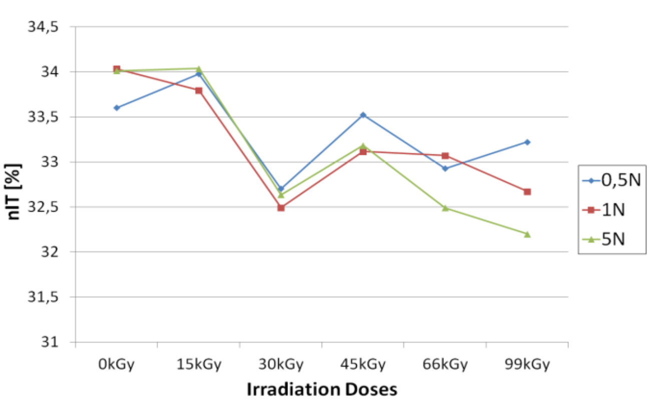

Fig. 26 Elastic part deformation work of PA6 vs. irradiation dose

E. *Creep behaviour*

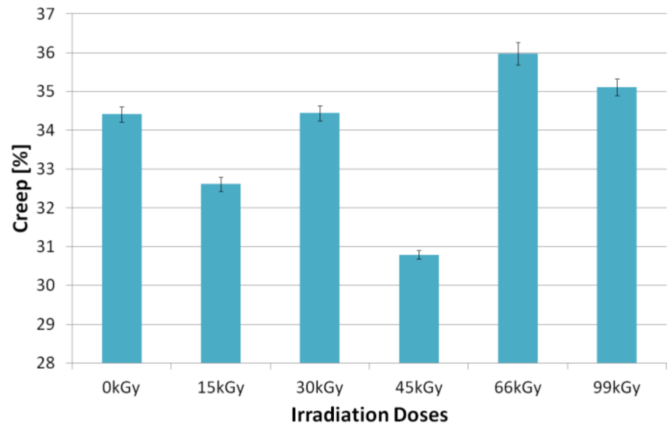

Fig. 27 Creep of PA6 vs. irradiation doses

Very important values were found for indentation creep. The lowest value of creep was measured at radiation dose of 45 kGy. The highest creep value measured at radiation dose of 66 kGy for polyamide 6. Decrease in creep values was 16% for irradiated polyamide 6 compared to the non-irradiated one as is seen at Fig. 27 and Fig. 28.

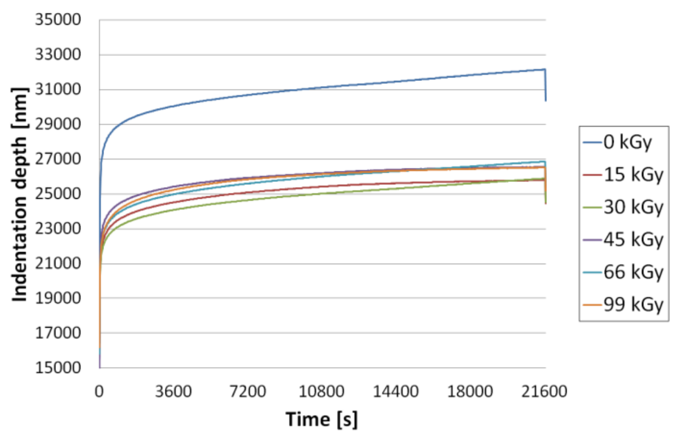

Fig. 28 Creep of PA6

IV. CONCLUSION

The properties of surface layer of polyamide 6 modified by beta radiation improved significantly. The microhardness values increased by about 41%. Stiffness of surface layer increased significantly by 50% as a result of radiation. The creep values decreased by 16% on average for irradiated polyamide . 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. The highest values of micromechanical properties were reached at radiation dose of 30 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 polyamide 6 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. The resistance of surface layer to wear makes its use suitable for the production of gears, friction parts of machinery and as alternative to some metal materials. Thanks to its low weight polyamide 6 modified by beta radiation is a suitable alternative to commonly used materials in the car and electrical industry.

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