Microhardness of Electron Beam Irradiated Polycarbonate

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

Abstract— Experimental study deals with the influence of beta radiation on the micromechanical properties of the surface layer of polycarbonate. When subjecting the polycarbonate to beta radiation, the structure as well as the surface layer show structural changes (crosslinking, degradation). The injected specimens were irradiated by doses of 0, 33, 66, 99, 132, 165 and 198 kGy. Irradiation of the specimens caused changes in the surface layer similar to the process of nitriding or cementation in metals. There was an improvement in some micromechanical properties of the surface layer of polycarbonate. The improvement/deterioration of micromechanical properties of surface layer of polycarbonate measured by the instrumented microhardness test is the content of this experimental study.

Keywords—Crosslinking, irradiation, microhardness, polycarbonate.

I. INTRODUCTION

Practical applications for radiation processing of materials have been evolving since the introduction of this technology more than 50 years ago. The main applications are for modification of polymer materials through radiation crosslinking, degradation and grafting. Radiation processing, using gamma-rays, electron-beams (EB), UV and X-rays or laser beams has been demonstrated on a large commercial scale to be very effective means of improving end-use properties of various polymers.

Modifications in chemical structure of plastic materials can be brought about either by conventional chemical means, usually involving silanes or peroxides, or by exposure to ionizing radiation from either radioactive sources, or highly accelerated electrons. Gamma-induced modifications in polycarbonate (PC) have been studied, in the dose range of 10×10^6 Gy. Investigations with Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction analysis (XRD) have been performed, indicating that at the dose of 10 MGy, phenolic group forms through the cleavage of ester bonds. The scission of the polymeric chain initiates a different morphological zone within the polymeric matrix, and the polymer becomes more crystalline with increasing dose.

Paper [1] investigated high-energy (MeV) - heavy ion irradiation of PC to study both chain scission and cross-linking. UV spectroscopy was used to verify the chemical changes induced by ^{7}Li and ^{59}Ni radiations. Chain scission has occurred for PC irradiated with ^{7}Li at the fluence of 1012 (ions/cm^2), while the end linking has taken place at the higher fluence of 5×10^{12} (ions/cm^2), and similarly with ^{59}Ni at 1012 (ions/cm^2).

Medical devices are some of the most demanding applications for the properties of polycarbonate. It shows very good physical and chemical properties, such as excellent transparency, light weight, heat resistance, stability and it is sterilizable by radiation methods [1]–[15].

The molecular weight (M_W) distribution of a material will also have a significant impact on its mechanical properties. The M_W of nylons is often measured by testing relative viscosity. Increasing relative viscosity, which occurs with increasing M_W, correlates with increasing impact toughness and is generally desirable. Increases in crystallinity also improve toughness by providing physical crosslinks. However, in both cases, there is some maximum M_W beyond which the effects become detrimental. This maximum depends on the mechanical property, the breadth of the M_W distribution, and the specific chemistry of the polymer [1]–[10].

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.

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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]–[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 (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 [12]–[24].

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%.
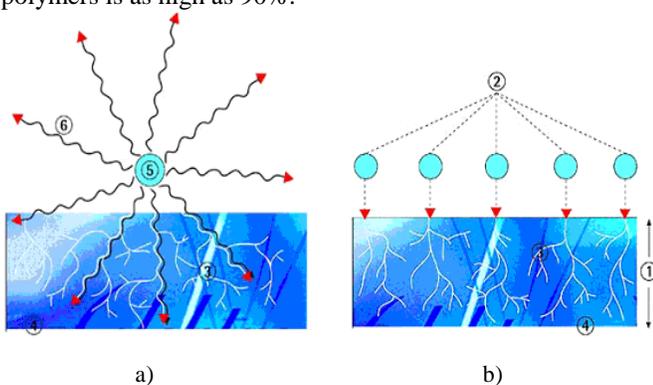
of all polymers [31]–[34].

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 micromechanical properties of PC.

II. EXPERIMENTAL

A. Irradiation

For this experiment polycarbonate PC Makrolon AL 2647, PTS-Plastics Technologie Service, Germany was used. The prepared specimens were irradiated with doses of 0, 33, 66, 99, 132, 165 and 198 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°).
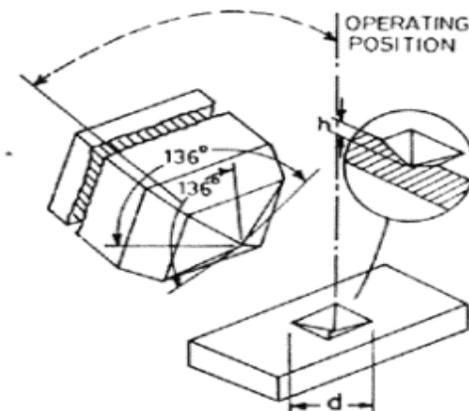

a)   b)

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

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 (Fig. 3), CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1. Load and unload speed was 1, 2 and 10 N/min. After a holding time of 90 s at

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 %

maximum loads 0.5 N, 1 N and 5 N the specimens were unloaded.

Fig. 3 Micro-combi tester

The indentation hardness H_{IT} was calculated as maximum load to the projected area of the hardness impression according to:

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

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 [5] [6].

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

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

The deduced modulus is calculated from the following equation:

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

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

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

Where E_i is the Elastic modulus of the indenter, E_r is the Reduced modulus of the indentation contact, v_i is the Poisson´s ratio of the indenter.

Determination of indentation hardness C_{IT}:

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

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 (Fig. 4) at time t_2 of holding the constant test force [1]–[5].
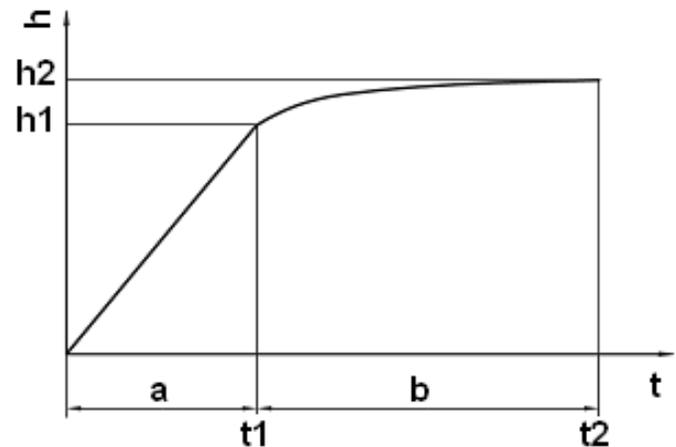

Fig. 4 Expression of indentation creep

Elastic part of the indentation work η_{IT}:

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

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

III. RESULTS AND DISCUSSION

A. Indentation load 0,5N

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the PC irradiated by a dose of 198 kGy. On the contrary, the highest values of indentation hardness were obtained for non-irradiated PC (by 11% higher in comparison with the PC irradiated by a dose of 198 kGy), as can be seen at Fig. 5.

Higher radiation dose does not influence significantly the Indentation hardness value. An indentation hardness increase of the surface layer is caused by irradiation cross-linking of the tested specimen.
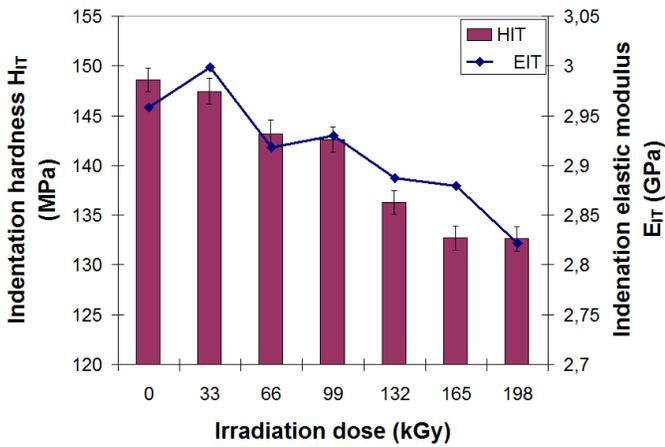
Fig. 5 Indentation hardness H_{IT} and indentation elastic modulus E_{IT} of PC vs. irradiation doses

A closer look at the microhardness results reveals that when the highest radiation doses are used, indentation hardness 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 non-irradiated PC irradiated with dose of 33 kGy (by 4% higher than compared with PC irradiated with dose of 198 kGy). On the contrary, the lowest values of the indentation modulus of elasticity were found for PC irradiated with dose of 198 kGy as is seen at Fig.5.

Fig. 6 Indentation creep of PC vs. irradiation doses

The lowest values of indentation creep were found for the PC irradiated by a dose of 165 kGy. On the contrary, the highest values of indentation creep were obtained for the PC irradiated by a dose of 165 kGy (by 7% higher in comparison with the PC irradiated by a dose of 165 kGy), as can be seen at Fig. 6.

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. 7).
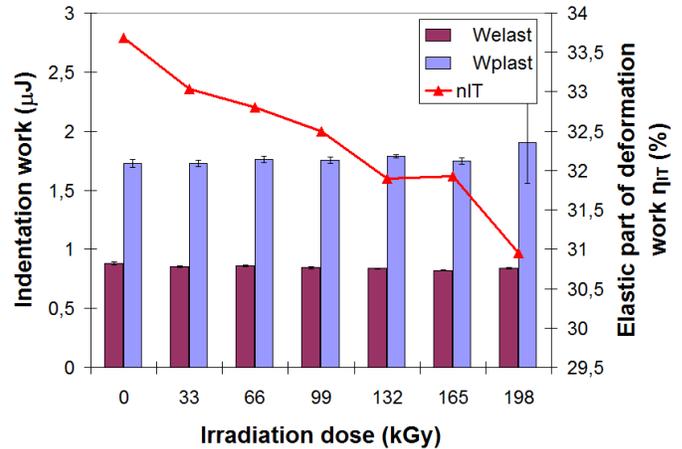

Fig. 7 Indentation work of PC vs. irradiation doses

B. Indentation load 1N

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the PC irradiated by a dose of 198 kGy. On the contrary, the highest values of indentation hardness were obtained for non-irradiated PC (by 11% higher in comparison with the PC irradiated by a dose of 198 kGy), as can be seen at Fig. 8.
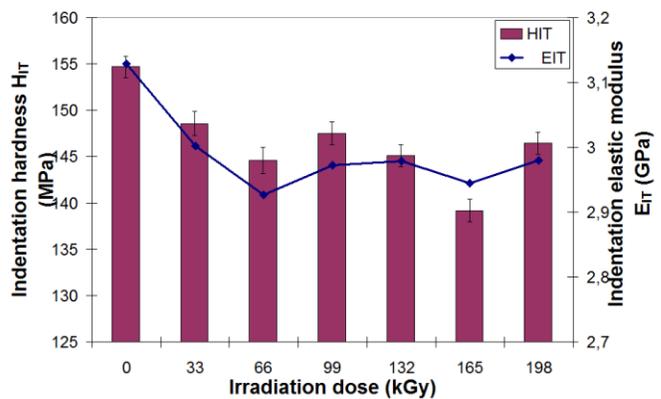

Fig. 8 Indentation hardness H_{IT} and indentation elastic modulus E_{IT} of PC vs. irradiation doses

Higher radiation dose does not influence significantly the indentation 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 indentation hardness results reveals that when the highest radiation doses are used, indentation hardness 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 non-irradiated PC (by 7% higher than compared with PC irradiated with dose of 66 kGy). On the contrary, the lowest values of the

indentation modulus of elasticity were found for PC irradiated with dose of 66 kGy as is seen at Fig.8.
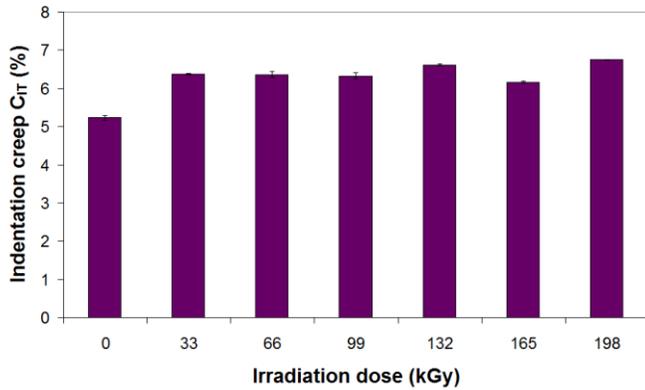

Fig. 9 Indentation creep of PC vs. irradiation doses

The lowest values of indentation creep were found for the non-irradiated PC. On the contrary, the highest values of indentation creep were obtained for the non-irradiated PC irradiated by dose 132 kGy (by 26% higher in comparison with the non-irradiated PC), as can be seen at Fig. 9.
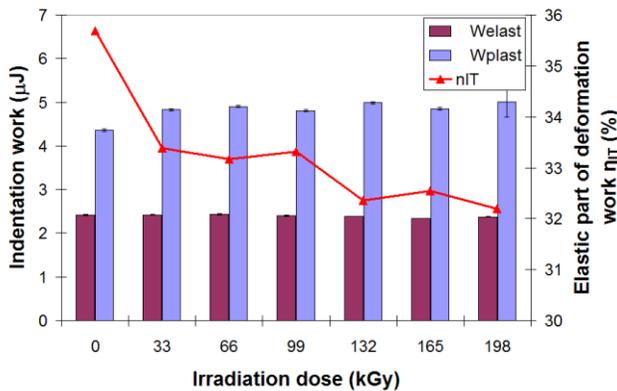

Fig. 10 Indentation work of PC vs. irradiation doses

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. 10).

C. Indentation load 5N

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the PC irradiated by a dose of 198 kGy. On the contrary, the highest values of indentation hardness were obtained for non-irradiated PC (by 11% higher in comparison with the non-irradiated PC), as can be seen at Fig. 11.
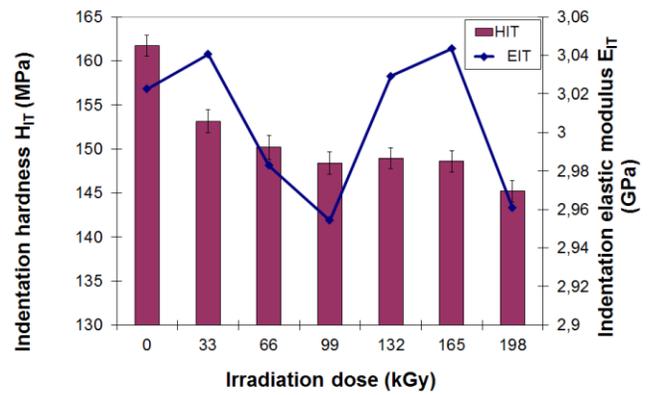

Fig. 11 Indentation hardness H_{IT} and indentation elastic modulus E_{IT} of PC vs. irradiation doses

Higher radiation dose does not influence significantly the indentation 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 indentation hardness results reveals that when the highest radiation doses are used, indentation hardness 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 PC irradiated with dose of 165 kGy (by 3% higher than compared with PC irradiated by 198 kGy). On the contrary, the lowest values of the indentation modulus of elasticity were found for PC irradiated by dose 198 kGy as is seen at Fig.11.

The lowest values of indentation creep were found for the PC irradiated by a dose of 99 kGy. On the contrary, the highest values of indentation creep were obtained for the PC irradiated 198 kGy (by 5% higher in comparison with the PC irradiated by a dose of 99 kGy), as can be seen at Fig. 12.
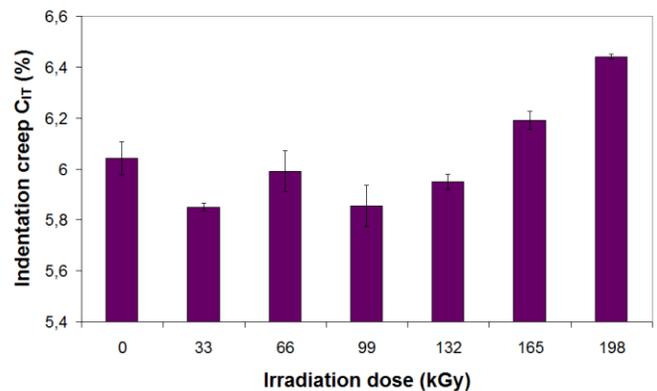

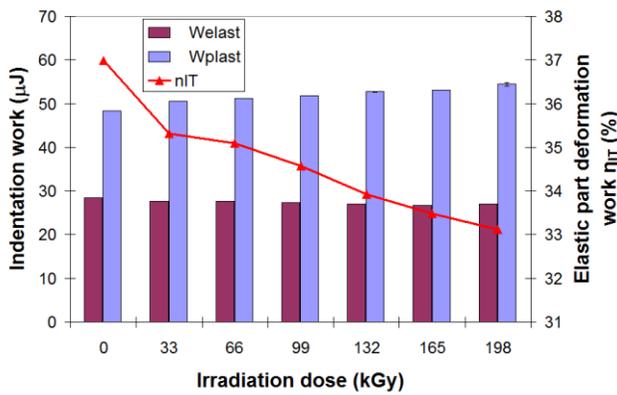Fig. 12 Indentation creep of PC vs. irradiation doses

Fig. 13 Indentation work of PC 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. 13).
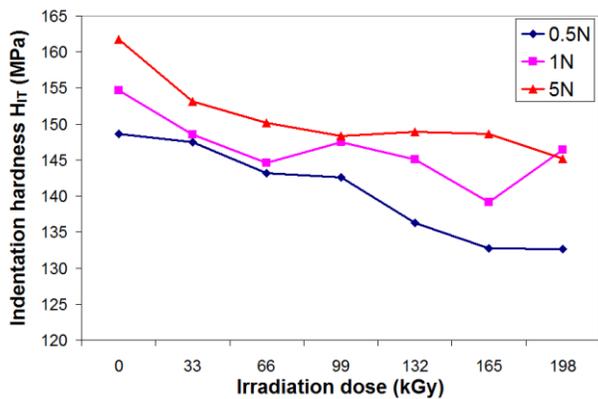

Fig. 14 Indentation hardness H_{IT} of PC vs. irradiation doses

Comparing the values of indentation hardness when applying different loads (0.5N, 1N a 5N) showed that the highest values were measured at 0.5N load. The lowest values of indentation hardness were found for the highest load of 5N. When applying the load of 0.5N, 1N and 5N the highest values of indentation hardness were measured at non-iraadiated PC. When applying the load (1N and 5N), the lowest value of indentation hardness was measured at the radiation dose of 198 kGy. When applying the load of 1N the lowest value of indentation hardness was measured at the radiation dose of 165 kGy (Fig. 14). These differences can be caused by the change of the structure towards the centre of the specimen. In fact the changes of the crosslinking values have a significant influence on the resulting values of indentation hardness.
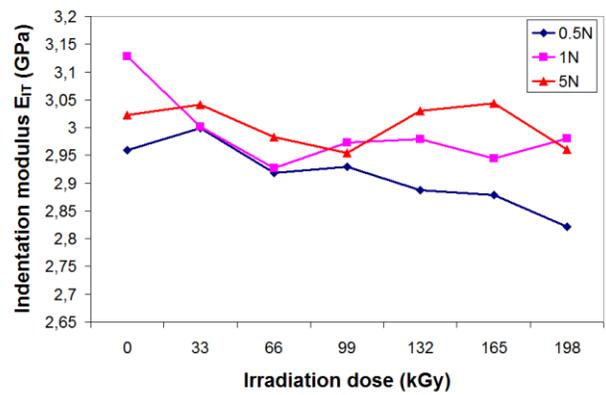

Fig. 15 Elastic modulus E_{IT} of PC vs. irradiation doses

The values of the elastic modulus when applying different loads (0.5N, 1N and 5N) show similar development. When examining the values of the elastic modulus, which characterizes the stiffness of the surface layer, it is clear that the maximum values were measured at the non-irradiated PC for all applied loads (0.5N, 1N and 5N). The lowest values were on the contrary measured at the PC irradiated 198 KGy by applied load 0,5N, 66 kGy by applied load 1N and 99 kGy by applied load 5N. When applying higher radiation doses (99 kGy) values show a slight decrease. This can be caused by structural changes such as degradation of the structure.
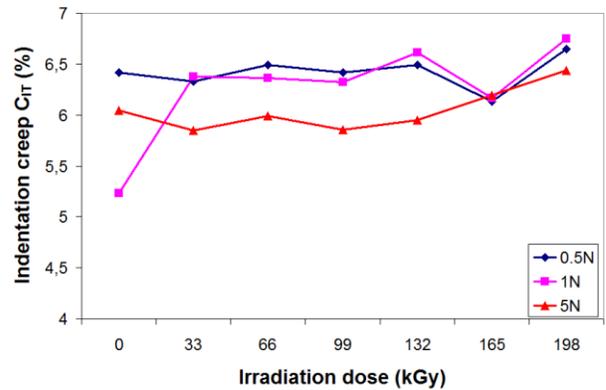

Fig. 16 Indentation creep of PC vs. irradiation doses

Creep behaviour of polymers is also a very important parameter. It indicates resistance of the surface layer of the specimen against constant deformation in the course of time. For irradiated polycarbonate the creep was measured at different loads of the specimen (0.1N, 1N and 5N). When applying the load of 0.5N it was found that the lowest creep values were measured at the radiation dose of 165 kGy. When applying the load of 1N it was found that the lowest creep values were measured at the non-irradiated PC. When applying the load of 5N it was found that the lowest creep values were measured at the radiation dose of 33 kGy. The highest values were found for the PC irradiated by dose 198 kGy for all

applied loads. Further drop in creep values did not occur with higher radiation doses, on the contrary there was a slight increase.
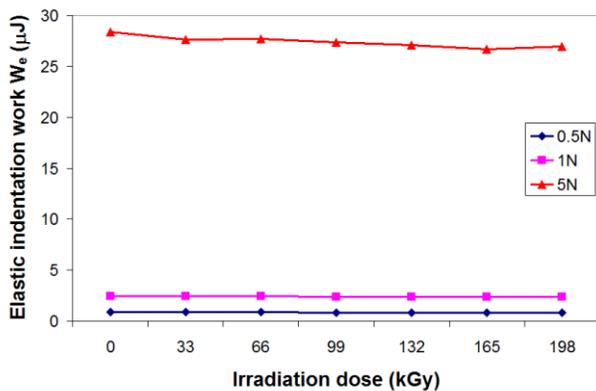

Fig. 17 Elastic indentation work of PC vs. irradiation doses

Instrumented microhardness test showed interesting results in respect of the work used at the test. Elastic and plastic indentation work gives us further information about the behaviour of the surface layer tested of the irradiated and non-irradiated polycarbonate. The results of the measurements show that the lowest values of elastic work were found in the case of specimen subjected to the radiation dose of 165 kGy. The highest values of the elastic work were measured for non-irradiated polycarbonate. The lowest values were found for specimen subjected to the non-irradiated PC, at the loads of 0.5N, 1N and 5N. The highest values of plastic indentation work were measured for the PC irradiated by dose 198 kGy. Elastic part of deformation work which characterizes the degree of recovery after indentation was again the highest at non-irradiated testing sample of PC and the lowest at radiation dose of 198 kGy .
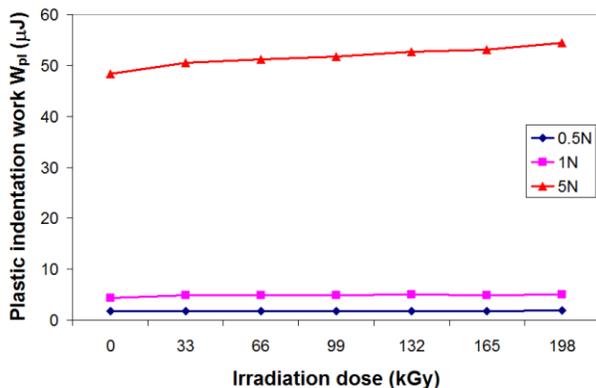

Fig. 18 Plastic indentation work of PC vs. irradiation doses

The figure 19 and 20 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 PC also proved very interesting. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens. The irradiated material showed low hardness as well as increasing impression of the indentor in the surface layer. On the contrary, the non-irradiated PC showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.
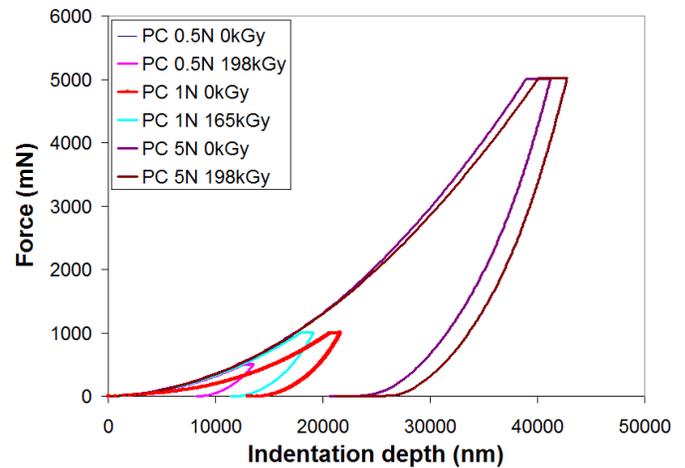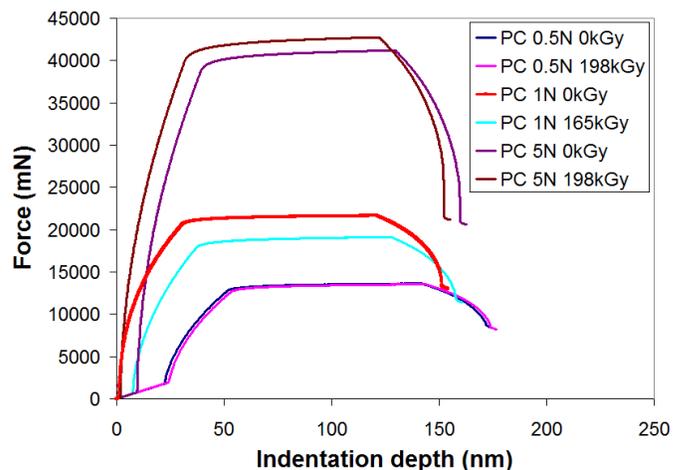

Fig. 19 Indentation depth vs. time

Fig. 20 Force vs. indentation depth

IV. CONCLUSION

The presented experimental study deals with the influence of beta irradiation on the properties of the surface layer of polycarbonate. The injected specimens were subjected to radiation dose of 0, 33, 45, 66, 99, 132, 165 and 198 kGy. The loads applied during the instrumented test of microhardness were 0.5N, 1N and 5N. The measurements were put in a graph

and evaluated.

The results show that the irradiation of the tested specimens results in worse micromechanical properties of polycarbonate. Indentation hardness values of the surface layer tested showed drop in values by 11% as a result of beta irradiation (at the radiation dose of 198 kGy). Elastic modulus, which characterizes the stiffness of the surface layer, showed drop in values by 7% at the radiation dose of 99 kGy. Creep showed drop in values by 5% at the radiation dose of 165 kGy compared to the non-irradiated PC. Beta irradiation process doesn´t have positive effect on the micromechanical properties of polycarbonate's surface layer.

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