Microhardness of Electron Beam Irradiated Polyamide 6.6

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 polyamide 6.6 with 6% crosslinking agent addition. When subjecting the polyamide 6.6 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, 45, 66 and 99 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 polyamide 6.6. The improvement/deterioration of micromechanical properties of surface layer of polyamide 6.6 measured by the instrumented microhardness test is the content of this experimental study.

Keywords—*Crosslinking, irradiation, microhardness, polyamide 6.6.*

I. Introduction

Nylon 6.6, or polyamide 6.6 (PA6.6), is a semicrystalline, thermoplastic commodity polymer that finds widespread use in applications that require considerable strength but low toughness [1]–[4].

The mechanical properties of PA6.6 can be dramatically influenced by the morphology of the final part, which is in turn influenced by the manufacturing and operating conditions. In the most stable PA6.6 crystalline structure, the triclinic α-phase, crystallites are comprised of stacks of crystalline. The sheets are formed by individual PA6.6 molecules hydrogen-bonded in all-trans conformations. These sheets are not rectangular, but have a slight shear due to a progressive offset between adjacent PA6.6 chains. Adjacent sheets are also regularly and cumulatively offset, tilting 42° relative the lamellar plane, resulting in a "pleated" crystallite. The second crystalline phase found in PA6.6, the β-phase, is also triclinic and characterized by sheets that shear alternately in the c-direction, resulting in a 13° offset from the lamellar normal. A third, nonequilibrium crystal phase is observed in PA6.6 at temperatures near the melting point. The γ-phase, or pseudohexagonal phase, begins to form when PA6.6 is heated through the "Brill transition." For PA6.6, the Brill transition temperature (T_B) occurs between 170 °C and 220 °C, well below the melting temperature (Tm), which is in the range of 250 °C to 272 °C. Any change in crystal structure would be expected to have an effect on physical behaviors including toughness and tensile strength.

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 MW 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]–[12].

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

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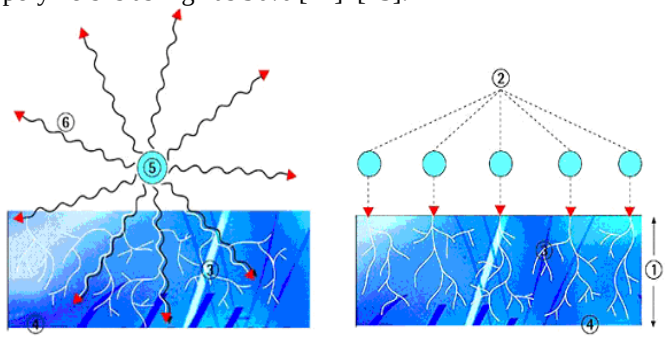
electrons emerge via a window (Titanium foil which occludes the vacuum) and are projected onto the product [3]–[9].

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 [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% [11]–[25].



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]–[12].

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 PA6.6, 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 PA6.6 structure. The utility properties of PA6.6 improve when the noncrystalline part of PA6.6 is cross-linked.

The present experimental work deals with the influence of beta irradiation on the microhardness of PA6.6.

II. Experimental

A. Irradiation

For this experiment polyamide PA6.6 V-PTS – Creamid-B3H2, PTS-Plastics Technology Service, Germany (unfilled, PA6.6+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[30]–[31].

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°)[25]–[31].

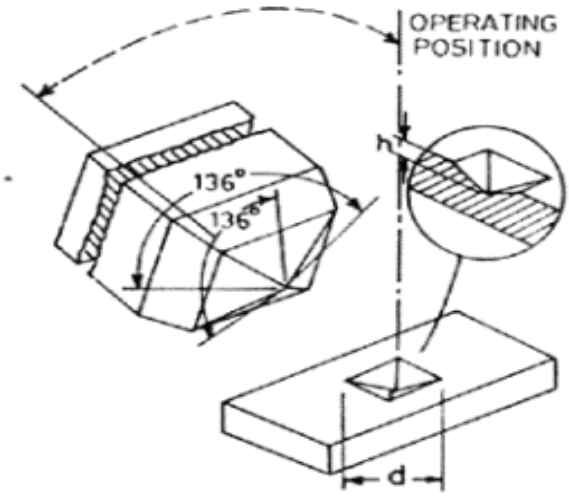


Fig. 2 The basic principle of hardness testing according to Vickers

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 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:

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

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:

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

The deduced modulus is calculated from the following equation:

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

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

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

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.

Determination of indentation hardness CIT:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100 \tag{5}$$

Where h1 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] [4] [5].

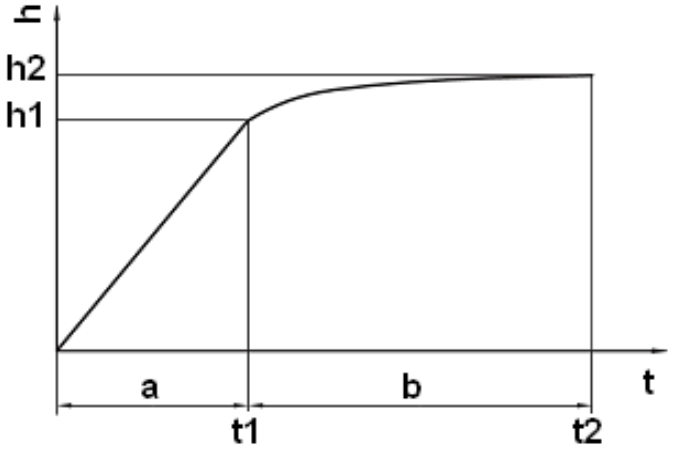


Fig. 4 Expression of indentation creep

Elastic part of the indentation work ηIT:

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

Plastic part W_{plast} / W_{total} follows as 100% - ηIT (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 non-irradiated PA6.6. On the contrary, the highest values of indentation hardness were obtained for PA6.6 irradiated by a dose of 45 kGy (by 7% higher in comparison with the non-irradiated PA6.6), 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. 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.

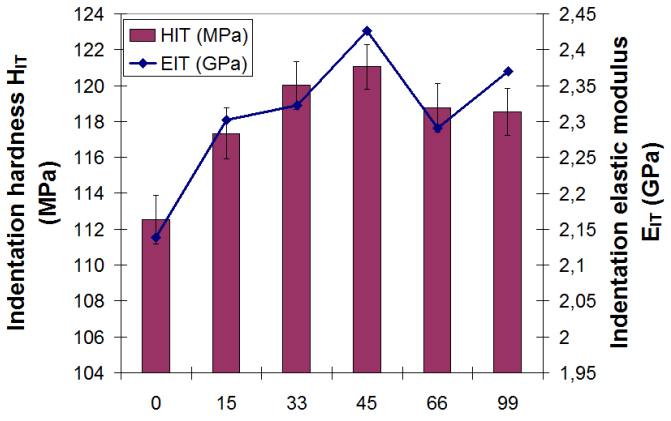


Fig. 5 Hardness H_{IT} and indentation elastic modulus E_{IT} of PA6.6 vs. irradiation doses

According to the results of measurements of microhardness, it was found that the highest values of indentation modulus of elasticity were achieved at the PA6.6 irradiated with dose of 45 kGy (by 13% higher than compared with non-irradiated PA6.6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6.6 as is seen at Fig. 5.

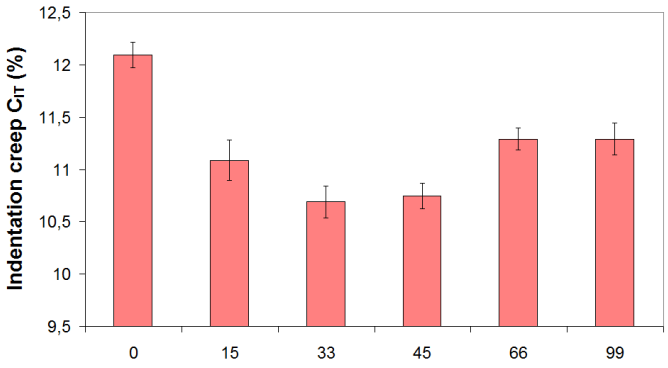


Fig. 6 Indentation Creep C_{IT} of PA6.6 vs. irradiation doses

The lowest values of indentation creep were found for the PA66 irradiated by a dose of 45 kGy. On the contrary, the highest values of indentation creep were obtained for the non-irradiated PA6.6 (by 8% higher in comparison with the PA6.6 irradiated by a dose of 45 kGy), as can be seen at Fig. 6.

Other important material parameters obtained during the microhardness test were elastic and plastic indentation work. The elastic deformation work W_e determines the reaction of material to applied (multiaxial) load with reversible deformation. The plastic part of the indentation work W_{pl} defines toughness of the tested material (surface layer) and its resistance to plastic deformation (Fig. 7).

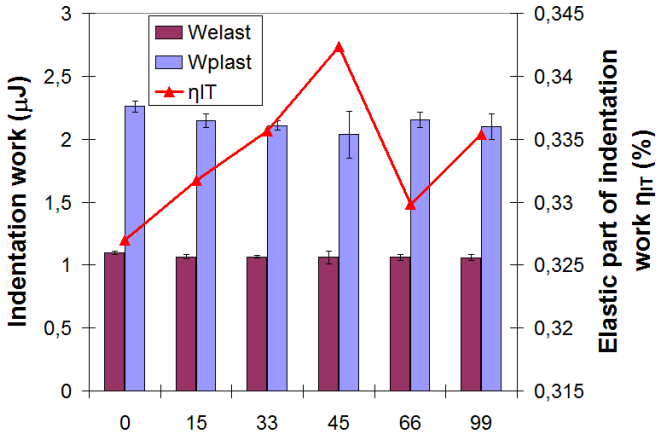


Fig. 7 Indentation work of PA6.6 vs. irradiation doses

B. *Indentation load 1N*

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated PA6.6. On the contrary, the highest values of indentation hardness were obtained for PA6.6 irradiated by a dose of 45 kGy (by 9% higher in comparison with the non-irradiated PA6.6), as can be seen at Fig. 8.

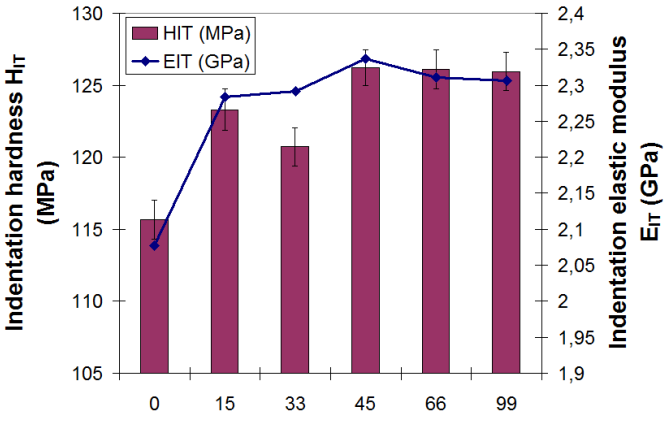


Fig. 8 Hardness H_{IT} and indentation elastic modulus E_{IT} of PA6.6 vs. irradiation doses

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 PA6.6 irradiated with dose of 45 kGy (by 12% higher than compared with non-irradiated PA6.6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6.6 as is seen at Fig. 8.

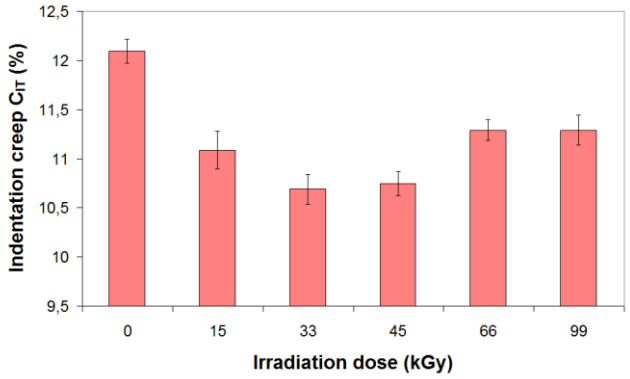


Fig. 9 Indentation Creep C_{IT} of PA6.6 vs. irradiation doses

The lowest values of indentation creep were found for the PA66 irradiated by a dose of 45 kGy. On the contrary, the highest values of indentation creep were obtained for the non-irradiated PA6.6 (by 12% higher in comparison with the PA6.6 irradiated by a dose of 45 kGy), as can be seen at Fig. 9.

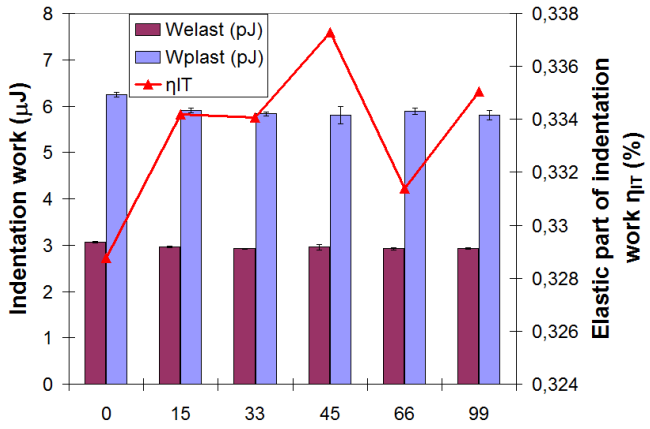


Fig. 10 Indentation work of PA6.6 vs. irradiation doses

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).

A. *Indentation load 5N*

The values measured during the microhardness test showed that the lowest values of indentation hardness were found for the non-irradiated PA6.6. On the contrary, the highest values of indentation hardness were obtained for PA6.6 irradiated by a dose of 45 kGy (by 9% higher in comparison with the non-irradiated PA6.6), as can be seen at Fig. 11.

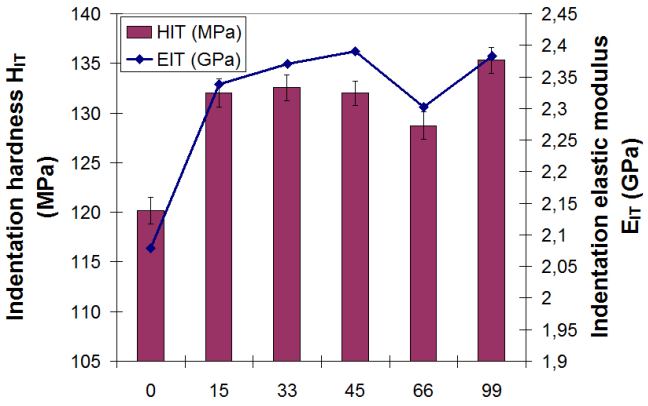


Fig. 11 Hardness H_{IT} and indentation elastic modulus E_{IT} of PA6.6 vs. irradiation doses

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 PA6.6 irradiated with dose of 45 kGy (by 15% higher than compared with non-irradiated PA6.6). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PA6.6 as is seen at Fig. 11.

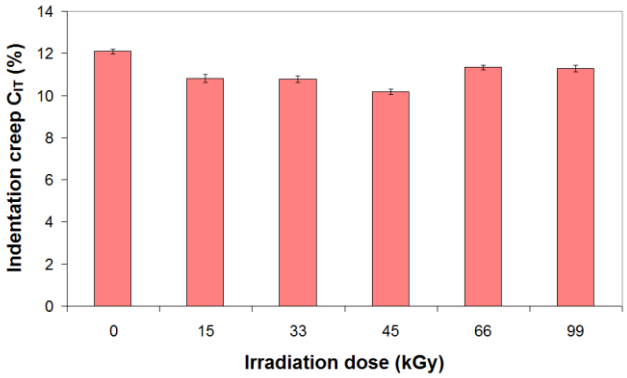


Fig. 12 Indentation Creep C_{IT} of PA6.6 vs. irradiation doses

The lowest values of indentation creep were found for the PA66 irradiated by a dose of 45 kGy. On the contrary, the highest values of indentation creep were obtained for the non-irradiated PA6.6 (by 16% higher in comparison with the

PA6.6 irradiated by a dose of 45 kGy), as can be seen at Fig. 12.

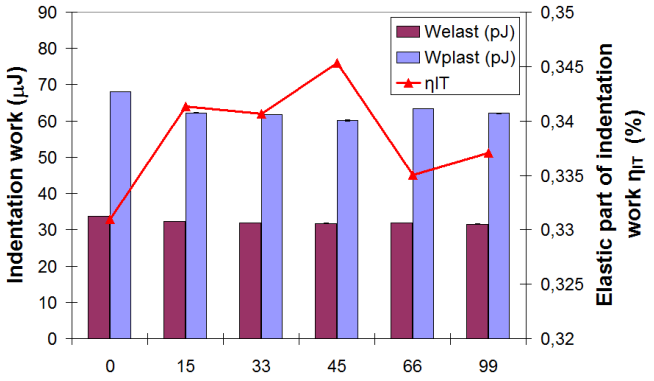


Fig. 13 Indentation work of PA6.6 vs. irradiation doses

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).

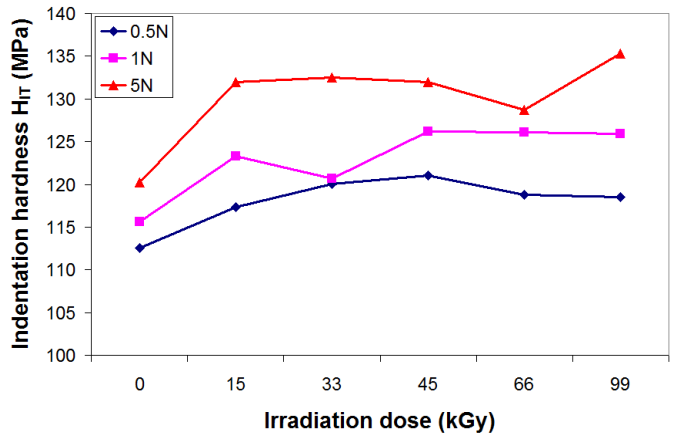


Fig. 14 Indentation hardness of PA6.6

Comparing the values of indentation hardness when applying different loads (0.5, 1 a 5N) showed that the highest values were measured at 5N load (Fig. 14). The lowest values of indentation hardness were found for the lowest load of 0.5N. When applying the load of 0.5 and 1 N the highest values of indentation hardness were measured at the radiation dose of 45 kGy. When applying the highest load (5N), the highest value of indentation hardness was measured at the radiation dose of 99 kGy. 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 and the amount of the crystalline and noncrystalline phases have a significant influence on the resulting values of microhardness.

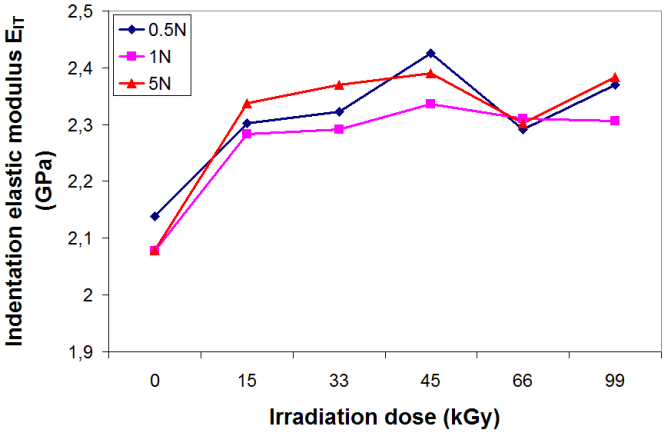


Fig. 15 Indentation elastic modulus of PA6.6

The values of the indentation elastic modulus when applying different loads (0.5N, 1N and 5N) show similar development. When examining the values of the indentation elastic modulus, which characterizes the stiffness of the surface layer, it is clear that the maximum values were measured at the radiation dose of 45 kGy for all applied loads (0.5N, 1N and 5N). The lowest values were on the contrary measured at non-irradiated specimens. When applying higher radiation doses (99kGy) values show a slight decrease. This can be caused by structural changes such as degradation of the structure (Fig. 15).

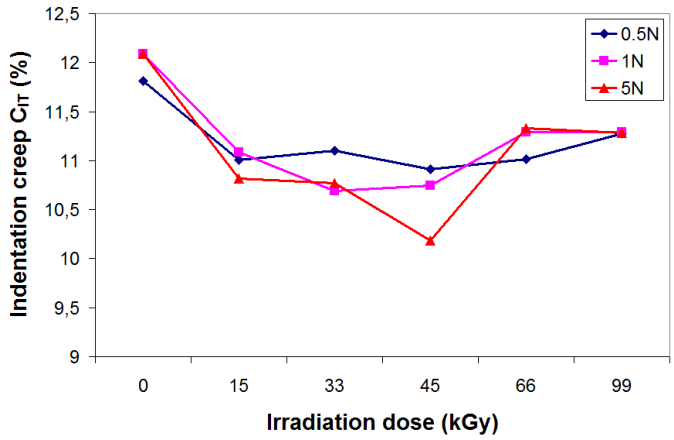


Fig. 16 Indentation creep of PA6.6

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 polyamide the creep was measured at different loads of the specimen (0.5N, 1N and 5N). The resulting values show that creep behaviour corresponds with the values of the elastic modulus. When applying the load of 0.5N, 1N and 5N it was found that the lowest creep values were measured at the radiation dose of 45 kGy. The highest values were found for non-irradiated PA6.6. Further drop in creep values did not

occur with higher radiation doses, on the contrary there was a slight increase (Fig. 16).

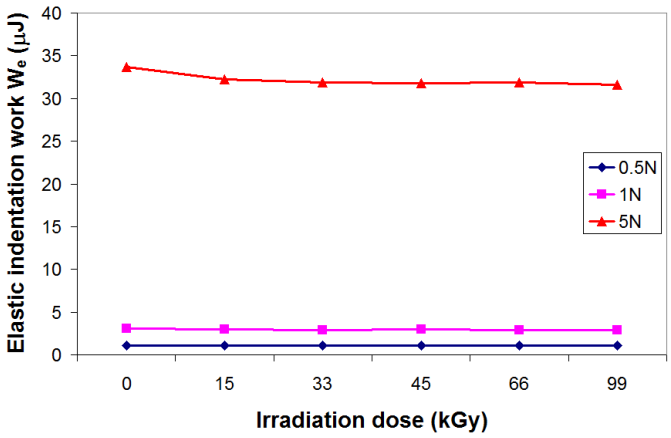


Fig. 17 Elastic indentation work of PA6.6

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 polyamide 6.6. 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 99kGy. The highest values of the elastic work were measured for non-irradiated polyamide 6.6. In the case of plastic work, the results correspond with some micromechanical properties such as creep or elastic modulus. The lowest values were found for specimen subjected to the radiation dose of 45 kGy, at the load of 0.5N, 1N and 5N. The highest values of plastic work were measured for non-irradiated polyamide 6.6. Elastic part of deformation work which characterizes the degree of recovery after indentation was again the highest at radiation dose of 45 kGy and the lowest at non-irradiated specimen (Fig. 17 and Fig. 18).

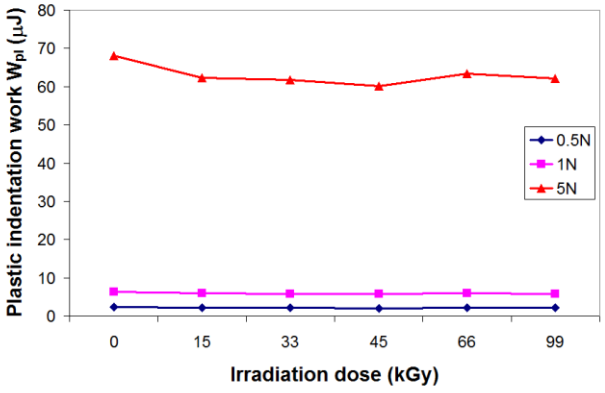


Fig. 18 Plastic indentation work of PA6.6

The figure 19 and 20 show 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 PA6.6 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 PA6.6 showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.

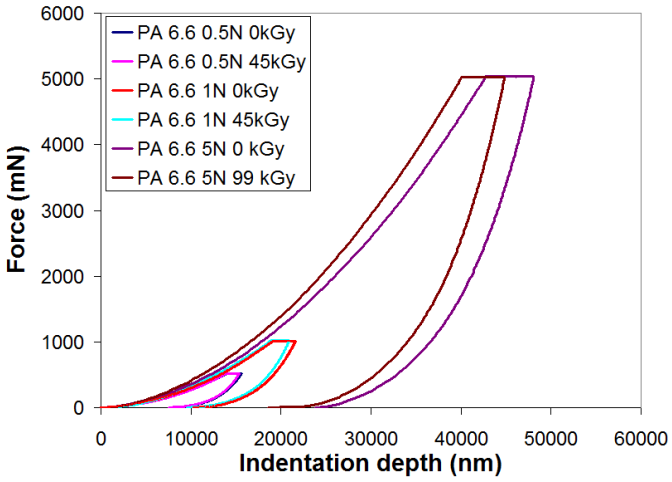


Fig. 19 Indentation depth vs. time

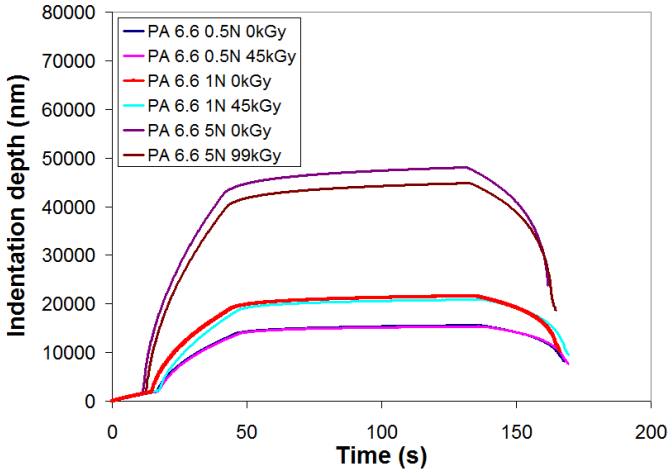


Fig. 20 Force vs. Indentation depth

IV. Conclusion

The presented experimental study deals with the influence of beta irradiation on the properties of the surface layer of polyamide 6.6. with 6% cross-linking agent addition. The injected specimens were subjected to radiation dose of 0, 33, 45, 66 and 99 kGy. The loads applied during the instrumented test of microhardness were 0.5, 1 and 5N. The measurements

were put in a graph and evaluated.

The results show that the irradiation of the tested specimens results in better micromechanical properties of polyamide 6.6. Microhardness values of the surface layer tested improved by 9% as a result of beta irradiation (at the radiation dose of 45 kGy). Elastic modulus, which characterizes the stiffness of the surface layer, improved by 15% at the radiation dose of 45 kGy. Creep showed drop in values by 16% at the radiation dose of 45 kGy compared to the non-irradiated PA 6.6.

Better micromechanical properties of the surface layer as a result of beta irradiation enable to use products made from polyamide 6.6 for more demanding applications. It is in fact the behaviour of the surface layer which plays a significant part in their application in the industry. The resistance of the surface layer against tear bite wears and deformations when subjected to long-term stress can have a considerable influence on the life cycle of the components used.

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