Micromechanical Properties of Surface Layer of HDPE Modified by Beta Irradiation

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Abstract — The experimental study deals with the effect of modification of the surface layer by irradiation cross-linking on the microchanical properties of the high-density polyethylene (HDPE) tested using the instrumented nanohardness test. The surface layer of HDPE specimen made by injection technology was modified by irradiation cross-linking using beta irradiation, which significantly influences micromechanical 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 micromechanical changes of the surface layer are observed in the instrumented nanohardness test.. The subject of this research is the influence of irradiation dosage on the changes of micromechanical properties of surface layer of HDPE.

Keywords—HDPE, nanohardness, irradiation crosslinking, surface layer, β – radiation.

I. INTRODUCTION

High density polyethylene (HDPE) has a melting point of 120 ± 130 _C and latent heat of fusion is as high as 180 ± 210 J/g. It has been reported that the melting point of HDPE is especially suitable for its use as a thermal energy storage material for solar absorption air conditioning

HDPE can be crosslinked by chemical or irradiation methods.

This paper is supported by the internal grant of TBU in Zlin No. IGA/FT/2014/016 funded from the resources of specific university research and by the European Regional Development Fund under the project CEBIA-Tech No. CZ.1.05/2.1.00/03.0089.

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Since crosslinked HDPE has a thermally stable form, it may be utilized as a thermal energy storage material in direct contact with the heat transferred, ethylene glycol. This thermally stable form of HDPE does not require separate packaging which increases the cost of the thermal energy storage system. Thus HDPE has been recommended as an economical thermal energy storage material with its large heat of fusion, relatively low cost and congruent melting behavior [1] [2] [3] [4] [5] [6] [7] [8] [9] [10].

Degradation is the major problem in the development of HDPE as a thermal energy storage material. Especially thermal oxidative degradation of HDPE produces low molecular weight products and oxygenated products, which have effects on melting point, heat of fusion and crystallinity. As thermal oxidative degradation proceeds, the amount of the degraded products becomes larger and the crystallinity of HDPE decreases, and hence the performance of the thermal energy storage system declines. In order to use HDPE as a thermal energy storage material, it should not be degraded during the period of application.

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.

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 [11] [12] [13] [14] [15] [16] [17] [18] [19].

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.

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 [20] [21] [22] [23] [24] [25] [26] [27] [28].

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%.



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.

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

Irradiation of HDPE by β -rays produces free radicals by kicking H-atoms, resulting in different chemical reactions, such as recrystallization, creation of hydroperoxides, cross-linking and degradation. The atmospheric oxygen diffusion in

HDPE sample during β -irradiation would form hydroperoxides at the polymer chains in the amorphous regions. By increasing the irradiation dose, cross-linking together with increasing of crystallinity in the amorphous regions is induced, and consequently the created hydroperoxides are predominant, resulting a reduction in the rate of degradation [29] [30] [31] [32] [33] [34].

The present experimental work deals with the influence of beta irradiation on the micromechanical properties of surface layer of HDPE.

II. EXPERIMENTAL

A. Nano-hardness according to Vickers

Test of hardness according to Vickers is prescribed by European standard CSN EN ISO 14577-1.

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' nanohardness 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°)[38] [39] [40] [41] [42] [43].



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

B. Material samples

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

C. Testing samples preparation

The samples were made using the injection molding

technology on the injection moulding machine Arburg Allrounder 420C. Processing temperature 200–240 °C, mold temperature 40 °C, injection pressure 80 MPa, injection rate 55 mm/s.

D. Instrumented nanohardness tests

Instrumented nanohardness tests were done using a Nanoindentation Tester (NHT2) – Opx/Cpx, CSM Instruments (Switzerland) according to the CSN EN ISO 14577-1. Load and unload speed was 20, 100 and 500 mN/min. After a holding time of 90 s at maximum load 10, 50 and 250 mN the specimens were unloaded. The indentation hardness H_{IT} was calculated as maximum load to the projected area of the hardness impression according to [44].

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

$$H_{IT} = \frac{F_{\max}}{A_p}$$
 with $h_c = h_{\max} - \varepsilon \frac{F_{\max}}{S}$ (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 [35] [36] [37].

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

$$E_{IT} = E * (1 - v_s^{2})$$
⁽²⁾

The deduced modulus is calculated from the following equation:

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

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

$$E^* = \frac{1}{\frac{1}{E_r} - \frac{1 - v_i^2}{E_i}}$$
(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 creep C_{IT}:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100$$
(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 [1] [4] [5].



Fig. 3 Expression of indentation creep Elastic part of the indentation work ηIT:

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

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

III. RESULTS AND DISCUSSION

A. Indentation load 10 mN

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found for the non-irradiated HDPE. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 99 kGy (by 25% higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 4.



Fig. 4 Hardness HIT of HDPE vs. irradiation doses

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

According to the results of measurements of nanohardness, it was found that the highest values of indentation modulus of elasticity were achieved at the HDPE irradiated with dose of 99 kGy (by 9% higher than compared with irradiated HDPE). On the contrary, the lowest values of the indentation modulus of elasticity were found for HDPE irradiated by a dose of 66 kGy as is seen at Fig. 5.



Fig. 5 Elastic modulus EIT of HDPE vs. irradiation doses

According to the results of measurements of nanohardness, it was found that the lowest values of indentation creep were achieved at the HDPE irradiated with dose of 99 kGy (by 19% lower than compared with non-irradiated HDPE). On the contrary, the highest values of the indentation creep were found for non-irradiated HDPE as is seen on Fig. 6.



Fig. 6 Indentation creep CIT of HDPE vs. irradiation doses

Other important material parameters obtained during the microhardness test were elastic and plastic deformation work. The elastic deformation work W_e determines the reaction of a

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.

The highest values of plastic deformation work were obtained for non-irradiated HDPE. The highest values of elastic deformation work were obtained for non-irradiated HDPE irradiated with a dose of 99 kGy. The lowest values of plastic deformation work were obtained for HDPE irradiated with a dose of 99 kGy. The lowest values of elastic deformation work were obtained for non-irradiated HDPE.

Next to plastic and elastic deformation work, the coefficient of back deformation η_{TT} is especially important for the assessment of the structure of the irradiated HDPE. The highest values were measured at irradiation doses of 199 kGy. The smallest values were found at non-irradiated PA12.

Interesting results were found for elastic and deformation work. The lowest value of elastic work was measured for irradiated HDPE by a dose 33kGy while the lowest value of plastic deformation work was found at the radiation dose of 99 kGy. The highest value at both deformation works was found when the lowest value of radiation dose of 0 kGy was applied (Fig. 7). Also, the value of elastic part of indentation work η_{IT} which provides information about the relaxation of the indent created in HDPE was the smallest for non-irradiated HDPE.



Fig. 7 Deformation work of HDPE vs. irradiation doses

B. Indentation load 50 mN

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found for the non-irradiated HDPE. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 99 kGy (by 13 % higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 8.





Fig. 8 Hardness H_{IT} of HDPE vs. irradiation doses

The highes values of indentation elastic modulus were found for the non-irradiated HDPE. On the contrary, the lowest values of indenation elastic modulus were obtained for HDPE irradiated by a dose of 66 kGy (by 10 % lower in comparison with the non-irradiated HDPE), as can be seen at Fig. 9.



Fig. 9 Elastic modulus E_{IT} of HDPE vs. irradiation doses

According to the results of measurements of nanohardness, it was found that the lowest values of indentation creep were achieved at the HDPE irradiated with dose of 99 kGy (by 3 % lower than compared with non-irradiated HDPE). On the contrary, the highest values of the indentation creep were found at the HDPE irradiated with dose of 33 kGy as is seen on Fig. 10.



Fig. 10 Indentation creep CIT of HDPE vs. irradiation doses

Interesting results were found for elastic and deformation work. The lowest value of elastic work was measured for non-irradiated HDPE, while the lowest value of plastic deformation work was found at the radiation dose of 99 kGy. The highest value of elastic deformation work was found for non-irradiated HDPE, and the highest value of plastic deformation work was found for non-irradiated HDPE too (Fig. 11). Also, the value of elastic part of indentation work η_{IT} which provides information about the relaxation of the indent created in HDPE was the smallest for non-irradiated HDPE.



Fig. 11 Deformation work of HDPE vs. irradiation doses

C. Indentation load 250 mN

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found at the HDPE irradiated by a dose 66 kGy. On the contrary, the highest values of indentation hardness were obtained for HDPE irradiated by a dose of 99 kGy (by 10 % higher in comparison with the non-irradiated HDPE), as can be seen at Fig. 12.



Fig. 12 Hardness H_{IT} of HDPE vs. irradiation doses

The highes values of indentation elastic modulus were found for the non-irradiated HDPE. On the contrary, the lowest values of indentaion elastic modulus were obtained for HDPE irradiated by a dose of 66 kGy (by 8 % lower in comparison with the non-irradiated HDPE), as can be seen at Fig. 13.



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

According to the results of measurements of nanohardness, it was found that the lowest values of indentation creep were achieved at the HDPE irradiated with dose of 33 kGy (by 4 % lower than compared with non-irradiated HDPE). On the contrary, the highest values of the indentation creep were found at the HDPE irradiated with dose of 99 kGy as is seen on Fig. 14.



Fig. 14 Indentation creep C_{IT} of HDPE vs. irradiation doses

The highest values of plastic deformation work were obtained for non-irradiated HDPE. The highest values of elastic deformation work were obtained for HDPE irradiated with a dose of 99 kGy. The lowest values of elastic deformation work were obtained for non-irradiated HDPE. The lowest values of plastic deformation work were obtained for HDPE irradiated with a dose of 199 kGy.

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



Fig. 15 Deformation work of HDPE vs. irradiation doses

The correlation between the force and the depth of the indentation in HDPE also proved very interesting (Fig. 15). It demonstrated the influence of radiation on the change in the mechanical properties of 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 HDPE showed considerably smaller depth of the impression of the indentor which can signify greater resistance of this layer to wear.



Fig. 15 Deformation work of HDPE vs. irradiation doses

Comparing the values of indentation hardness when applying different loads (10 mN, 50 mN a 250 mN) showed that the highest values were measured at 5N load (Fig. 16). The lowest values of indentation hardness were found for the lowest load of 250 mN. When applying the load of 10 mN and 50mN the highest values of indentation hardness were measured at the radiation dose of 99 kGy. When applying the highest load (250 mN), the highest value of indentation hardness was measured at the radiation dose of 99 kGy. This fact 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 nanohardness.



Fig. 17 Deformation work of HDPE vs. irradiation doses

The values of the indentation elastic modulus when applying different loads (10 mN, 50 mN and 250 mN) 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 99 kGy for all applied loads (10 mN, 50 mN and 250 mN). The lowest values were on the contrary measured at non-irradiated specimens. When applying higher radiation doses (99kGy) values show a slight increase. This can be caused by structural changes such as crosslinking of the structure (Fig. 17).



Fig. 17 Deformation work of HDPE 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 HDPE the creep was measured at different loads of the specimen (10 mN, 50 mN and 250 mN). The resulting values show that creep behaviour corresponds with the values of the elastic modulus. When applying the load of 50 mN and 250 mN it was found that the lowest creep values were measured at the radiation dose of 99 kGy. The highest values were found for non-irradiated HDPE irradiated by a dose 66 kGy (10 and 50mN). Further drop in creep values did not occur with higher radiation doses, on the contrary there was a slight increase (Fig. 18).



Fig. 18 Deformation work of HDPE vs. irradiation doses

IV. CONCLUSION

Very interesting results were obtained for irradiation modified HDPE. When comparing the irradiated and nonirradiated HDPE it was apparent that the values of indentation hardness, elastic modulus considerably increased, in some cases even by 25% at the irradiation dose of 99kGy. Also different depths of indentation in the surface layer of tested specimen were significantly different. 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.

Improvement of mechanical properties in micro and macro scale of radiated HDPE has a great significance also for industry. The modified polypropylene shifts to the group of materials which have considerably better properties. Its heat and micromechanical properties make HDPE ideal for a wide application in the areas where higher resistance to wear, creep and higher temperatures are required. Commonly manufactured HDPE can hardly fulfill these criteria. Thanks to its low weight HDPE modified by beta radiation is a suitable alternative to commonly used materials in the car and electrical industry.

ACKNOWLEDGMENT

This paper is supported by the internal grant of TBU in Zlin No. IGA/FT/2014/016 funded from the resources of specific university research and by the European Regional Development Fund under the project CEBIA-Tech No. CZ.1.05/2.1.00/03.

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