Micro-Hardness of PBT Influenced by Beta Radiation

Martin Ovsik[1,a], Lenka Hylova[1], David Manas[1], Miroslav Manas[1] and Michal Stanek[1]

[1] *Tomas Bata University in Zlin, TGM 5555, 760 01 Zlin, Czech Republic*

Abstract. Cross-linking is a process in which polymer chains are associated through chemical bonds. Radiation, which penetrated through specimens and reacted with the cross-linking agent, gradually formed cross-linking (3D net), first in the surface layer and then in the total volume, which resulted in considerable changes in specimen behaviour. This research paper deals with the utilization of electron beam irradiated PBT on the micro-indentation test. Radiation doses of 66, 132 and 198 kGy were used for unfilled PBT with the 5% crosslinking agent (triallyl isocyanurate). Individual radiation doses caused structural and micro-mechanical changes which have a significant effect on the final properties of the PBT tested. The highest values of micro-mechanical properties were reached radiation dose of 132 kGy, when the micro-mechanical values increased by about 30%.

1 Introduction

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

PBT are characterized by their high strength and rigidity, dimensionally stable, low tendency to creep, very good frictional and wear resistance, good impact strength, very low coefficient of thermal expansion, good chemical resistance to acids, very good electrical characteristics, very low moisture absorption, good adhesion and welding ability. Furthermore, PBT, like all polyesters, has very good frictional and wearing properties. Compared to PET, PA6 has a better impact strength - particularly at low temperatures [2-4].

Cross-linking is a process in which polymer chains are associated through chemical bonds. Cross-linking is carried out by chemical reactions or radiation and in most cases the process is irreversible. Ionizing radiation includes high-energy electrons (electron beam - β-rays) (Fig. 1) and gamma rays (γ-rays) (Fig. 2). These not only are capable of converting monomeric and oligomeric liquids into solids, but also can produce major changes in properties of solid polymers. [3]

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

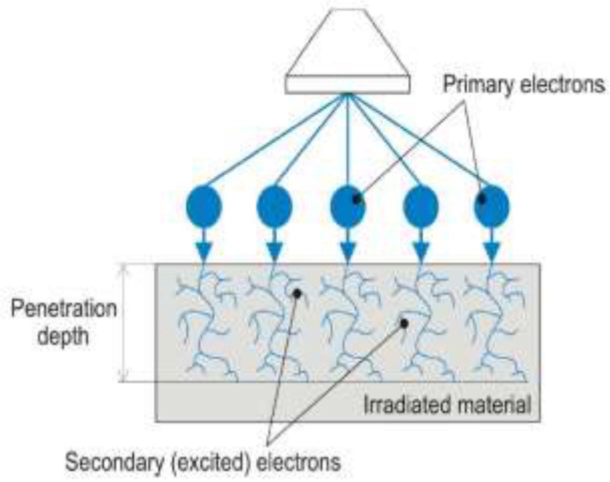

Figure 1. Radiation crosslinking by electrons rays

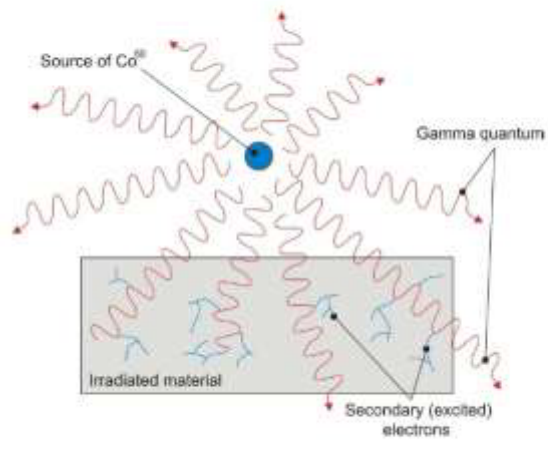

Figure 2. Radiation crosslinking by Gamma rays

[a] Corresponding author: ovsik@ft.utb.cz

The thermoplastics which are used for production of various types of products have very different properties. Standard polymers which are easy obtainable with favorable price conditions belong to the main class. The disadvantage of standard polymers is limited both by mechanical and thermal properties. The group of standard polymers is the most considerable one and its share in the production of all polymers is as high as 90% [1] [5].

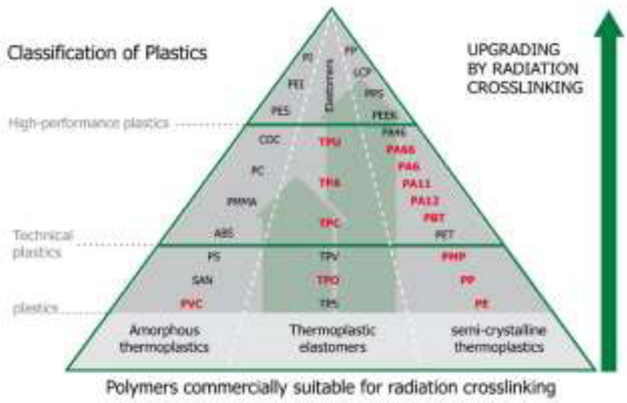

Figure 3. Upgrading properties by radiation cross-linking

The aim of this paper is to study the effect of ionizing radiation with different doses, on micro-mechanical properties of PBT and compare these results with those of non-irradiated samples. These tested polymers find a broad application in many branches of industry and in daily life as well.

2 Experimental

2.1 Material

For this experiment Polybutylene terephthalate PBT V-PTS-CREATEC-B3HZC * M800/25, PTS Plastics Technology Service, Germany, was used. The material already contained a special cross-linking agent TAIC (5 volume %), which should enable subsequent cross-linking by ionizing β – radiation.

2.2 Sample preparation

The samples were made using the injection molding technology on the injection molding machine Arburg Allrounder 470H. Processing temperature 225–255 °C, mold temperature 70 °C, injection pressure 90 MPa, injection rate 50 mm/s. It was used normalized specimen measuring 80x10x4 mm.

2.3 Irradiation

The prepared specimens were irradiated with doses of 0, 66, 132 and 198 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany.

2.4 Micro-indentation

Micro-indentation test were performed using a Micro-indentation tester (Micro Combi Tester), CSM Instruments (Switzerland) according to the CSN EN ISO 14577. The tip is made of diamond having the shape of a cube corner (Vickers). In the present study, the maximum load used was 0.5 N and loading rate (and unloading rate) was 1 N/min. A holding time was 90 s at the indentation and 21600 s at the creep.

Figure 4. Micro-indentation tester

The indentation hardness (H_{IT}) was calculated as maximum load (F_{max}) to the projected area of the hardness impression (A_p) and the indentation modulus (E_{IT}) is calculated from the Plane Strain modulus (E^*) using an estimated sample Poisson's ratio (ν) according to [5-7]:

$$H_{IT} = \frac{F_{\max}}{A_p} \quad (1)$$

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

Measurement of all above mentioned properties was performed 10 times to ensure statistical correctness.

3 Results and discussion

Radiation, which penetrated through specimens and reacted with the cross-linking agent, gradually formed cross-linking (3D net), first in the surface layer and then in the total volume, which resulted in considerable changes in specimen behaviour.

Micro-indentation characteristics determined by DSI method are depicted in Fig. 1 and 2. They characterize course of loading force in dependence on indentor penetration depth, which gives an idea about course of instantaneous values of observed micro-mechanical properties.

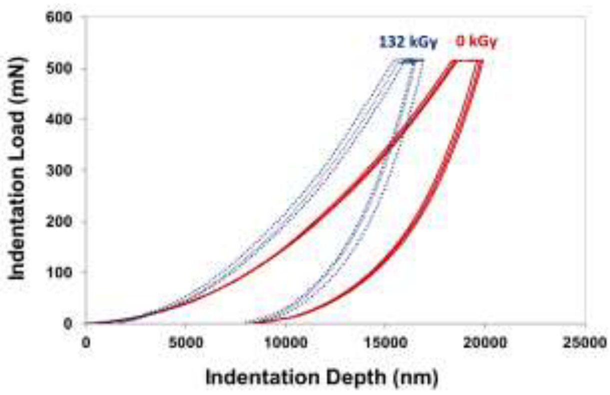

Figure 5. Indentation characteristic of irradiated PBT

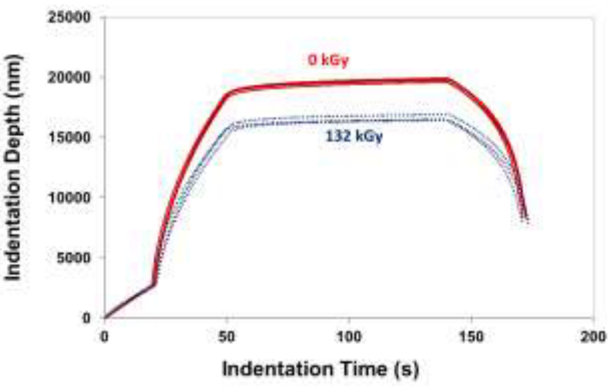

Figure 6. Indentation characteristic of irradiated PBT

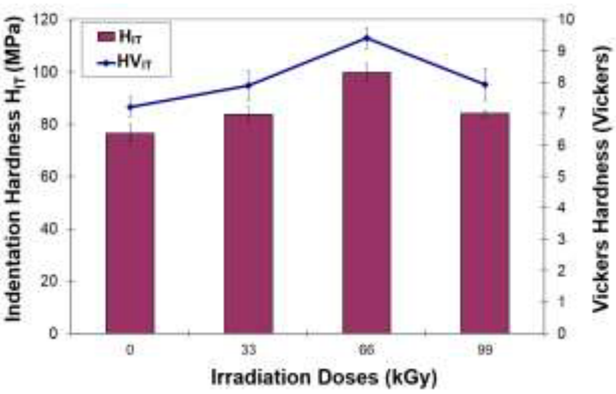

Figure 7. Indentation hardness ($H_{IT,}$ HV_{IT})

It demonstrated the influence of radiation on the change of micro-mechanical properties in the surface layer of specimens. The non-irradiated material showed low hardness as well as increasing impression of the indenter in the surface layer. On the contrary, the irradiated PBT showed considerably smaller depth of the impression of the indenter which can signify greater resistance of this layer to wear (Fig. 5 and Fig. 6).

The values measured during the micro-indentation test showed that the lowest values of indentation hardness and Vickers hardness (H_{IT} = 76 MPa, HV_{IT} = 7 Vickers) were found for the non-irradiated PBT. On the contrary, the highest values of indentation hardness and Vickers hardness (H_{IT} = 100 MPa, HV_{IT} = 9 Vickers) were obtained for PBT irradiated by a dose of 132 kGy (by 30% higher in comparison with the non-irradiated PBT), as can be seen at Fig. 7.

In the case of indentation modulus the highest value was found for PBT irradiated by the radiation dose of 132 kGy (E_{IT} = 1.8 GPa). The smallest value of indentation modulus was found for non-irradiated PBT (E_{IT} = 1.4 GPa). The increase of the value of PBT irradiated by the radiation dose of 132 kGy was by 31% in comparison to the non-irradiated PBT (Fig. 8).

Very important values were found for indentation creep (C_{IT}). The lowest value of indentation creep was measured at radiation dose of 132 kGy (C_{IT} = 14.7%). The highest indentation creep value measured at non-irradiated PBT (C_{IT} = 28.2%). Decrease in creep values was 92% for irradiated PBT compared to the non-irradiated one as is seen at Fig. 11.

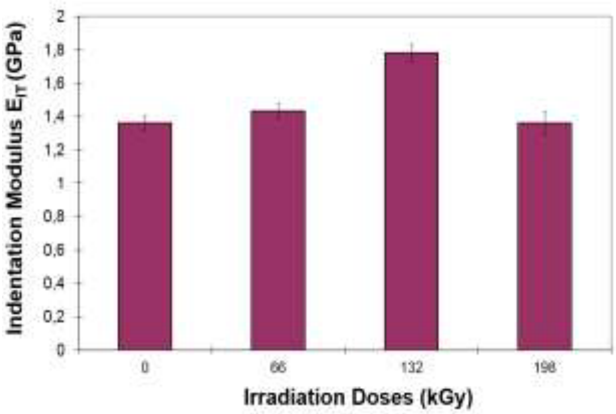

Figure 8. Indentation Modulus (E_{IT})

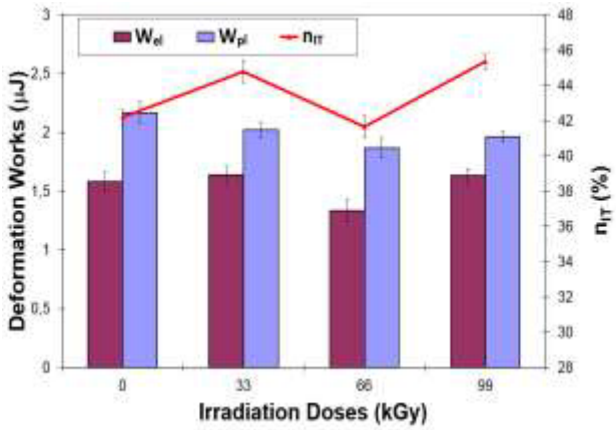

Figure 9. Elastic and plastic part of deformation work

Other important material parameters obtained during the micro-indentation test were elastic and plastic deformation work. The greatest values of plastic and elastic deformation work and coefficient of back deformation η_{IT} were obtained for non-irradiated PBT (W_{el} = 1.6 μJ, W_{pl} = 2.2 μJ, n_{IT} = 42.2 %,). The lowest values of W_{el}, W_{pl} and η_{IT} were obtained for PBT irradiated with dose of 132 kGy (Wel = 1.3 μJ, Wpl = 1.9 μJ, nIT = 41.6 %,). Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 9.

Radiation cross-linking creates changes in the PBT structure by creating 3D net. Beta radiation gradually penetrates more deeply into the PBT structure through the surface layer. The surface layer undergoes changes which have a considerable influence on the micro-mechanical properties of PBT.

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

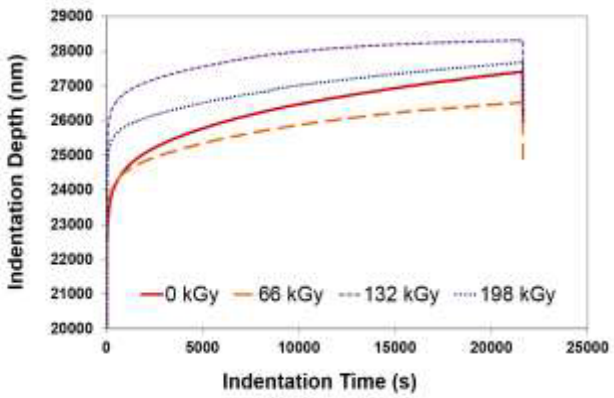

Figure 10. Indentation creep (C_{IT})

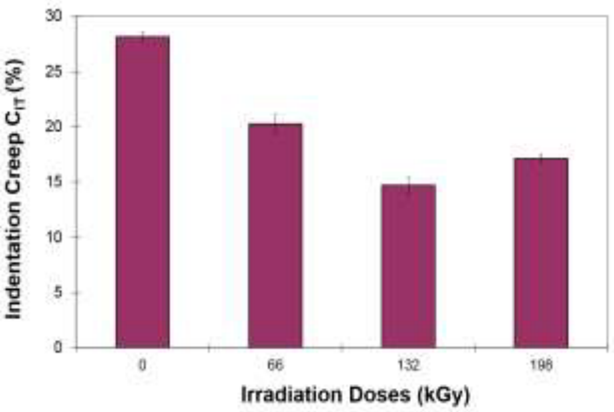

Figure 11. Indentation creep (C_{IT})

4 Conclusion

This research paper investigates influence of modified polymer material (beta radiation) on the micro-indentation test. The surface layer of PBT is modified by β – radiation with doses of 66, 132 and 198 kGy.

Radiation, which penetrated through specimens and reacted with the cross-linking agent, gradually formed cross-linking (3D net), first in the surface layer and then in the total volume, which resulted in considerable changes in specimen behaviour.

Irradiation of PBT with a β - radiation influences the micro-mechanical properties in the following way:

• Radiation of specimens caused improvement values of indentation hardness and indentation modulus.

• The highest values of indentation hardness and indentation modulus were achieved at the PBT irradiated with dose of 132 kGy (The micro-hardness values were increased by 30% and indentation modulus increased significantly by 31% as a result of radiation).

• Higher radiation dose does not influence the indentation hardness and indentation modulus significantly, on the contrary due to degradation processes the properties deteriorate.

• Values of indentation hardness and indentation modulus correspond to the deformation works.

The results of micro-mechanical properties of surface layer of modified PBT show that it can be used in more difficult applications in some industrial fields, in particular where there are high requirements for strength, stiffness and hardness of surface layer which appears to be the most suitable area of application

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