## LOCAL MECHANICAL PROPERTIES OF IRRADIATED CROSS-LINKED FILLED POLY (BUTYLENE TEREPHTHALATE) (PBT)

### LOKALNE MEHANSKE LASTNOSTI RADIOAKTIVNO OBSEVANEGA PREČNO VEZANEGA POLIBUTILEN TEREFTALATA (PBT), OJAČANEGA S STEKLENIMI VLAKNI

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This article deals with the measurement of the mechanical properties of a filled poly (butylene terephthalate) (PBT+35 % GF (glass fibres)) surface layer modified with beta radiation. Injection-moulded test bodies were subsequently irradiated with beta radiation using doses of (0, 33, 66 and 99) kGy. The measurement of the mechanical properties was realized with an ultra nano-hardness tester. The results of the measurements showed a considerable increase in the micromechanical properties (indentation hardness, indentation elastic modulus) when low doses of beta radiation were used. The aim of this paper is to study the effect of ionizing radiation at different doses on the ultra nano-hardness of the surface layer of poly (butylene terephthalate) (PBT) and compare these results with those for non-irradiated samples. The study was carried out due to the ever-growing use of this type of polymer, poly (butylene terephthalate) (PBT).

Keywords: poly (butylene terephthalate) (PBT+35 %GF), surface layer, mechanical properties, ultra nano-hardness

V članku avtorji opisujejo mehanske lastnosti s steklenimi vlakni ojačanega polibutilen tereftalata (PBT+35 % steklenih vlaken (GF)) po radioaktivnem obsevanju  $\gamma$ . Injekcijsko brizgani preizkušanci so bili obsevani z dozami (0, 33, 66 in 99) kGy radioaktivnega sevanja  $\gamma$ . Lokalne mehanske lastnosti na površini preizkušancev so določili z avtomatskim merilnikom ultra nanotrdote. Rezultati meritev so pokazali znatno povišanje mikromehanskih lastnosti (nanotrdote in elastičnega modula vtiskovanja) že pri nizkih dozah obsevanja vzorcev. Namen tega prispevka je prikazati vpliv ionizirajočega sevanja različnih jakosti na ultrananotrdoto površinskih plasti PBT+35% GF in primerjavo z neobsevanim vzorcem. Raziskava je bila izdelana zaradi vse večje uporabe tega polimernega materiala v pogojih ionizirajočega sevanja.

Ključne besede: polibutilen tereftalat, ojačan s 35 % steklenih vlaken (PBT+35 %GF); površinska plast, mehanske lastnosti, ultrananotrdota

#### 1 INTRODUCTION

Poly (butylene terephthalate) (PBT) is a versatile engineering thermoplastic, used for many industrial/commercial applications, which is often processed using injection moulding due to its favoured flow characteristics. With the melting temperature of around 220 °C and the glass-transition temperature of about 45 °C, this polymer is often found in electrical and automotive applications. Both the excellent physical properties and challenging design drawbacks of PBT are due to its inherent semicrystalline microstructure. During the injection moulding, this semicrystalline microstructure forms under shear and thermal gradients, typically leading to the development of variable morphologies between the skin and the core, with the subsequent implications on the property profile.<sup>1</sup>

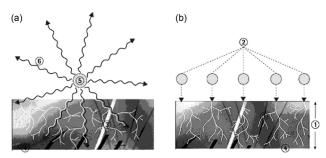
The irradiation cross-linking of thermoplastic materials via an electron beam or cobalt 60 (gamma rays) proceeds separately after the processing. The cross-link-

ing level can be adjusted with the irradiation dosage and often by means of a cross-linking booster.

The main deference between  $\gamma$ - and  $\gamma$ -rays (**Figure 1**) is in their different abilities of penetrating the irradiated material;  $\gamma$ -rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons.

Thermoplastics used for the production of various types of products have very different properties. Standard polymers that are easily obtainable at favourable prices belong to the main class. The disadvantage of standard polymers relates to both the 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 %.

Engineering polymers are a very important group of polymers, exhibiting much better properties in comparison to those of standard polymers. Both mechanical and thermal properties are much better than in the case of standard polymers. The production of these types of



**Figure 1:** a) design of gamma rays and b) electron rays: 3 – secondary electrons, 4 – irradiated material, 5 – encapsulated Co – 60 radiation source, 6 – gamma rays, b) 1 – penetration depth of electrons, 2 – primary electrons, 3 – secondary electrons, 4 – irradiated material

polymers accounts for less than 1 % of all the polymers.  $^{1\text{-}6}$ 

The present work deals with the influence of beta irradiation on the mechanical properties of the surface layer of injection-moulded filled PBT (PBT+35 % GF (glass fibres)).

#### 2 EXPERIMENTAL PART

For this experiment, polybutylene terephthalate (PBT + 35 % GF) V-PTS-CREATEC-B3HG7ZC \*M800/25 natur., PTS Plastics Technologie Service, Germany, was used. The material already contained a special crosslinking agent, TAIC – triallyl isocyanurate (6 volume %), which enabled the subsequent cross-linking by ionizing  $\gamma$ -radiation. Irradiation was carried out at the company BGS Beta Gamma Service GmbH & Co, KG, Saal an der Donau, Germany, using electron rays, an electron energy of 10 MeV, and doses of (0, 33, 66 and 99) kGy on air at the ambient temperature.

Samples (**Figure 2**) were made using the injection-moulding technology on an injection-moulding machine, Arburg Allrounder 420 °C. The processing temperature was 245–295 °C, the mould temperature was 85 °C, the injection pressure was 80 MPa and the injection rate was 45 mm/s.<sup>7-11</sup>

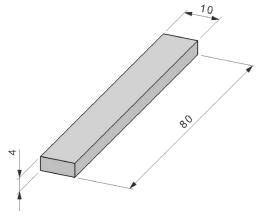


Figure 2: Dimensions of a sample

A nanoindentation test was done using an ultra nanoindentation tester (UNHT), CSM Instruments (Switzerland), according to CSN EN ISO 14577. The load and unload speed was 1000 N/min. After a holding time of 90 s at the maximum load of 500  $\mu$ N, the specimens were unloaded. The specimens were glued onto metallic sample holders (**Figure 2**).<sup>7–11</sup>

$$H_{\rm IT} = F_{\rm max} / A_{\rm p} \tag{1}$$

Here  $H_{\rm IT}$  is the indentation hardness,  $F_{\rm max}$  is the maximum applied force,  $A_{\rm p}$  is the projected area of the contact between the indenter and the test piece determined from the force-displacement curve and the knowledge of the area function of the indenter.<sup>7-11</sup>

#### **3 RESULTS**

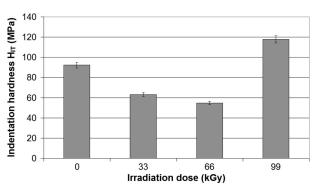


Figure 3: Indentation hardness  $H_{\rm IT}$ 

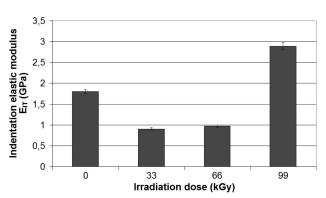


Figure 4: Indentation elastic modulus  $E_{\rm IT}$ 

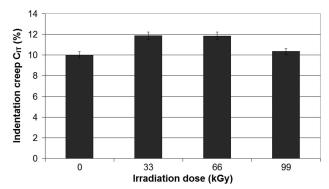


Figure 5: Indentation creep  $C_{\rm IT}$ 

#### 4 DISCUSSION

The development of the micromechanical properties of the irradiated PBT+35 %GF was characterized with an test of the ultra nanohardness ( $H_{\rm IT}$ ), as can be seen in **Figure 3**. The lowest value (54 MPa) of the indentation hardness was found on the PBT+35 %GF irradiated with a radiation dose of 66 kGy, while the highest value of the indentation hardness was found on the PBT+35 %GF irradiated with a radiation dose of 99 kGy (117 MPa). The increase of the indentation hardness at the 99 kGy radiation dose was 27 % compared to the non-irradiated PBT+35 %GF.

A similar development was recorded for the microstiffness of the specimens represented by the indentation elastic modulus ( $E_{\rm IT}$ ) illustrated in **Figure 4**. The results of the measurements show clearly that the lowest value of the indentation elastic modulus was measured on the PBT + 35 % GF (0.9 GPa) irradiated with the radiation dose of 33 kGy, while the highest value was found on the PBT + 35 % GF irradiated by 99 kGy (2.89 GPa). A significant increase in the indentation elastic modulus (60 %) was recorded at the radiation dose of 99 kGy compared to the non-irradiated PBT + 35 % GF.

Very important values were found for the indentation creep. For the materials, which creep as polymers, the basic calculation of that creep can be measured during a pause at the maximum force. The creep is the relative change of the indentation depth when the test force is kept constant. The measurements of the ultra nano-hardness obtained with the tests (**Figure 5**) showed that the highest creep value was measured on the sample irradiated with the 33 kGy dose (11.8 %), while the lowest creep value was found for the PBT + 35 % GF irradiated with the 0 kGy dose (10.02 %).

#### **5 CONCLUSIONS**

This article deals with the measurements of the mechanical properties of a poly (butylene terephthalate) (PBT) surface layer modified with beta radiation. Injection-moulded test bodies were irradiated with beta radiation using the doses of (0, 33, 66 and 99) kGy. The measurements of the mechanical properties were realized with an ultra nano-hardness tester.

The measurement results show an improvement in the chosen mechanical properties. The micro-hardness of the poly (butylene terephthalate) (PBT) sample surface layer irradiated with a 99 kGy dose of beta radiation increased by 27 %. The rigidity of the tested surface layer represented by the modulus of elasticity increased

by 60 % for the sample irradiated with a 99 kGy dose of beta radiation. The creep of the tested surface layer increased from 10.02 % for the non-irradiated sample to a value of 11.8 % for the sample irradiated with a 33 kGy dose of beta radiation.

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