

## LOCAL MECHANICAL PROPERTIES OF IRRADIATED CROSS-LINKED POLYPROPYLENE

### LOKALNE MEHANSKE LASTNOSTI RADIOAKTIVNO OBSEVANEGA PREČNO VEZANEGA POLIPROPILENA

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*Prejem rokopisa – received: 2017-07-01; sprejem za objavo – accepted for publication: 2017-10-11*

doi:10.17222/mit.2017.097

Using high doses of beta radiation for isotactic polypropylene (iPP) and its influence on the changes in the micromechanical properties of the surface layer has not been studied in detail so far. Specimens of isotactic polypropylene (iPP) were made with the injection-moulding technology and irradiated with high doses of beta radiation (0, 45, 66 and 99) kGy. The changes in the micromechanical properties of the surface layer were evaluated using an ultra nano-hardness test. The results of the measurements showed a considerable increase in the micromechanical properties (indentation hardness, indentation elastic modulus) when high doses of beta radiation are used. The aim of this paper is to study the effect of ionizing radiation with different doses on the ultra nanohardness of the surface layer of isotactic polypropylene (iPP) and compare these results with those of non-irradiated samples. The study was carried out due to the ever-growing use of this type of polymer, isotactic polypropylene (iPP).

Keywords: isotactic polypropylene (iPP), surface layer, mechanical properties, ultra nanohardness

Avtorji prispevka so raziskovali vpliv močnega radioaktivnega sevanja  $\beta$  na mikromehanske lastnosti površinskih plasti izotaktičnega polipropilena (iPP), kar do sedaj še ni bilo natančneje raziskano. Vzorci iPP so bili izdelani s tehnologijo injekcijskega brizganja in obsevani z visokimi dozami  $\beta$  radioaktivnega sevanja (0, 45, 66 in 99) kGy. Mikromehanske lastnosti površinskih plasti obsevanih vzorcev so določili z inštrumentiranim merilnikom ultranano-trdote. Rezultati meritev so pokazali znatno zvišanje mikromehanskih lastnosti (nanotrdo in modula elastičnosti) radioaktivno obsevanih vzorcev. Namen tega prispevka je prikazati vpliv radioaktivnega sevanja  $\beta$  različnih jakosti na površinske plasti iPP in primerjavo z neobsevanim vzorcem. Raziskava je bila izdelana zaradi vse večje uporabe tega polimernega materiala v pogojih radioaktivnega sevanja.

Ključne besede: polietilen z veliko gostoto (HDPE), površinska plast, mehanske lastnosti, ultranano-trdota

## 1 INTRODUCTION

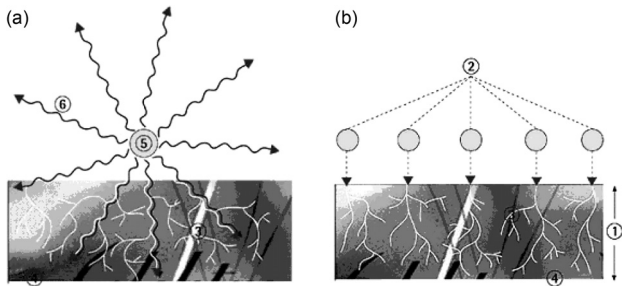
Isotactic polypropylene (iPP) is a commodity polymer with a semi-crystalline structure, which is very complex and depends strongly on the thermal history and processing conditions. Isotactic polypropylene can crystallize into three phases: the  $\alpha$ -phase is the most stable and the most common. The crystals are monoclinic. The  $\beta$ -phase is metastable and its crystals are hexagonal. The  $\beta$ -phase is mainly found in block PP copolymers and can be generated by adding specific nucleating agents. This phase was discovered by Padden and Keith in 1953 and can be improved with a crystallization between 130 °C and 132 °C or an orientation with high shear or through additions of specific nucleating agents. The presence of the  $\beta$ -phase in PP homopolymer generally increases the ductility of finished parts. The maximum effect is observed at 65 % of the  $\beta$ -phase. The  $\gamma$ -phase is also metastable, with triclinic crystals. This form is not very common; it appears mainly in low-molecular-weight polypropylene due to the crystallization at a very high pressure and very low cooling rate.<sup>1-2</sup>

The irradiation cross-linking of thermoplastic materials via an electron beam or cobalt 60 (gamma rays) proceeds separately after the processing. The cross-linking level can be adjusted with the irradiation dosage and often by means of a cross-linking booster.

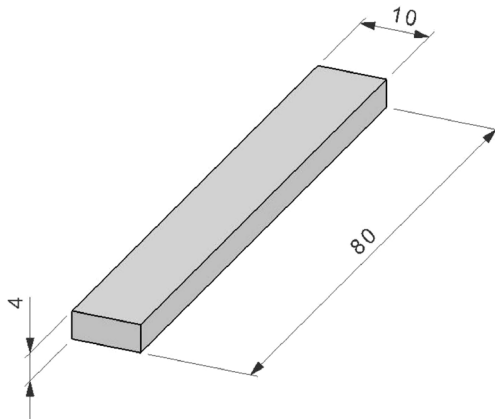
The main difference between  $\beta$ - and  $\gamma$ -rays (**Figure 1**) is in their different abilities of penetrating the irradiated material;  $\gamma$ -rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons.

Thermoplastics used for the production of various types of products have very different properties. Standard polymers that are easy obtainable at favourable price conditions 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 %.

The present work deals with the influence of beta irradiation on the mechanical properties of the surface layer of injection-moulded isotactic polypropylene (iPP).<sup>3-6</sup>



**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 –



**Figure 2:** Dimensions of sample

## 2 EXPERIMENTAL PART

For this experiment, PTS-Crealen EP-2300L1-M800, PTS Plastics Technologie Service, Germany (unfilled, iPP+TAIC, MFR–230 °C /2, 16 kg–6 g/10 min) was used. The material already contained a special cross-linking agent, TAIC – triallylisocyanurate (6 % of volume fractions), which enabled the subsequent cross-linking with ionizing  $\beta$ -radiation. Irradiation was carried out at the company BGS Beta-Gamma-Service GmbH & Co, KG, Germany, using electron rays, an electron energy of 10 MeV, and doses of (0, 45, 66 and 99) kGy in air at ambient temperature.

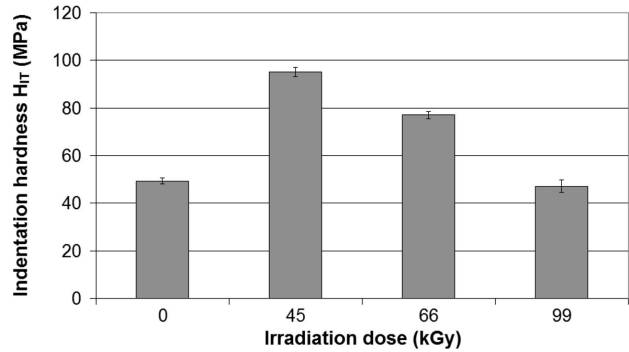
Samples (**Figure 2**) were made using the injection-moulding technology on an injection-moulding machine, Arburg Allrounder 420C. The processing temperature was 245–295 °C, the mould temperature was 85 °C, the injection pressure was 80 MPa and the injection rate was 45 mm/s.<sup>7–11</sup>

A nanoindentation test was done using an ultra nanoindentation tester (UNHT), CSM Instruments (Switzerland), according to the CSN EN ISO 14577. 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>

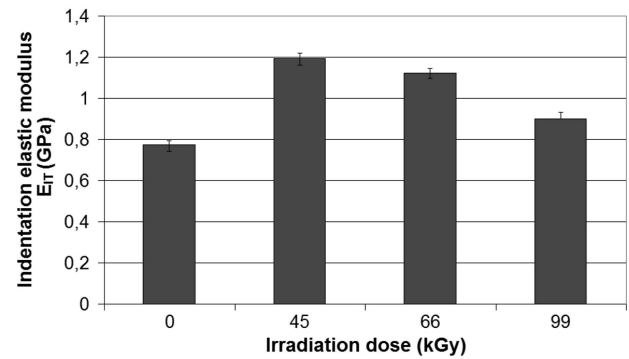
$$H_{IT} = F_{max}/A_p \quad (1)$$

Here  $H_{IT}$  is the indentation hardness,  $F_{max}$  is the maximum applied force, and  $A_p$  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>

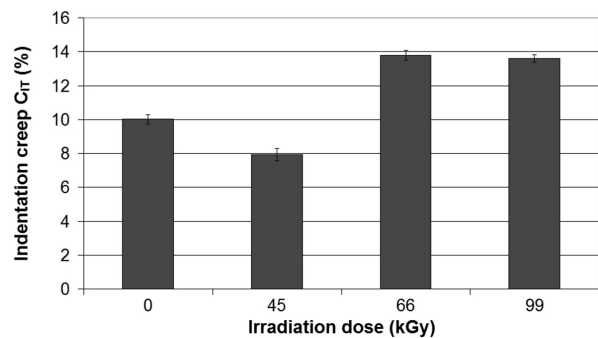
## 3 RESULTS



**Figure 3:** Indentation hardness  $H_{IT}$



**Figure 4:** Indentation elastic modulus  $E_{IT}$



**Figure 5:** Indentation creep  $C_{IT}$

## 4 DISCUSSION

The development of the micromechanical properties of the irradiated isotactic polypropylene (iPP) was characterized with a test of the ultra nano-hardness ( $H_{IT}$ ), as can be seen in **Figure 3**. The lowest value (47 MPa) of the indentation hardness was found for the isotactic polypropylene (iPP) irradiated with the dose of 99 kGy, while the highest value of the indentation hardness was

found for the isotactic polypropylene (iPP) irradiated with the dose of 45 kGy (95 MPa). The increase in the indentation hardness at the 45 kGy radiation dose was 92 %, compared to the non-irradiated isotactic polypropylene (iPP).

A similar development was recorded for the micro-stiffness of the specimens represented by the indentation elastic modulus ( $E_{IT}$ ) illustrated in **Figure 4**. The results of the measurements show clearly that the lowest value of the indentation elastic modulus was measured for the isotactic polypropylene (iPP) (0.77 GPa) irradiated with the dose of 0 kGy, while the highest value was found for the isotactic polypropylene (iPP) irradiated with 45 kGy (1.19 GPa). A significant increase in the indentation elastic modulus (54 %) was recorded at the radiation dose of 198 kGy, compared to the non-irradiated isotactic polypropylene (iPP).

Very important values were found for the indentation creep. For the materials, which creep as polymers, the basic calculation of the creep can be measured during a pause at the maximum force. The creep is a relative change of the indentation depth when the test force is kept constant. The measurements of the ultra nano-hardness showed (**Figure 5**) that the highest creep value was obtained for the sample irradiated with the 66 kGy dose (13.7 %), while the lowest creep value was found for the isotactic polypropylene (iPP) irradiated with the 45 kGy dose (7.9 %). The creep decreased by 21 % because of the radiation, which is a considerable increase in the surface-layer resistance.

## 5 CONCLUSIONS

This article deals with the measurements of the mechanical properties of the tested isotactic polypropylene (iPP) surface layer modified with beta radiation. Injection-moulded test bodies were irradiated with beta radiation using doses of (0, 45, 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 ultra nano-hardness of the isotactic polypropylene (iPP) surface layer irradiated with the 45 kGy dose increased by 92 %. The rigidity of the tested surface layer represented by the modulus of elasticity increased by 54 % for the sample irradiated with the dose of 45 kGy. The creep of the tested surface layer decreased from 10 % for the

non-irradiated sample to a value of 7.9 % for the sample irradiated with the dose 45 kGy.

## Acknowledgment

This paper was supported by an internal grant of the TBU in Zlin, No. IGA/FT/2017/010, funded from the resources for the specific university research and by the Ministry of Education, Youth and Sports of the Czech Republic within the National Sustainability Programme, project No. LO1303 (MSMT-7778/2014) and also by the European Regional Development Fund under project CEBIA-Tech, No. CZ.1.05/2.1.00/03.0089. Special thanks go to Dr. Michal Danek (BGS Beta-Gamma-Service GmbH & Co, KG, Germany) for his kind assistance during the sample preparation.

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