Influence of Irradiation on Mechanical Properties of PMMA

P. Kratky, D. Manas, M. Manas, M. Stanek, M. Ovsik, V. Senkerik, J. Navratil

***Abstract* —** The goal of the experimental study is clarify the effect of modification of the surface layer by irradiation on the mechanical properties of the PMMA tested using the instrumented nanohardness test. Surface layer was affected by radiation cross-linking technology which allows polymer materials modification followed by the change of their end-use properties. 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 mechanical 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 mechanical properties of PMMA.

***Keywords* —** Crosslinking, irradiated, nano-hardness, PMMA.

I. INTRODUCTION

Economical alternative to polycarbonate with lower strenght in other applications is polymethyl methacrylate which is frequently preferred because of its moderate properties, easy handling and processing, and low cost. Polymethyl methacrylate belongs to the group of synthetic thermoplastics polymers and is a transparent thermoplastic. It used as a lightweight or shatter-resistant alternative to glass.

As polymers belong to constructive materials which find use at the most industry branches. The advantage is a low weight together with the excellent mechanical properties, very good chemical resistance and other properties, which assign them for various applications. Disadvantage is mainly low temperature stability which significantly reduces usage of these polymers.

Every properties improvement especially temperature stability helps to increase application possibilities. In addition, properties modification of standard polymers, which are relatively cheap products, gives them advantage for another usage. One of the possibilities of polymers improvement is their radiation cross-linking.

The irradiation cross-linking of thermoplastic materials via electron beam or cobalt 60 (gamma rays) is performed separately, after processing. Generally, ionizing radiation includes accelerated electrons, gamma rays and X-rays.1

Radiation processing with an electron beam offers several distinct advantages when compared with other radiation sources, particularly γ-rays and x-rays. The process is very fast, clean and can be controlled with much precision. There is no permanent radioactivity since the machine can be switched off. In contrast to γ-rays and x-rays, the electron beam can be steered relatively easily, thus allowing irradiation of a variety of physical shapes. The electron beam radiation process is practically free of waste products and therefore is no serious environmental hazard. The main difference between beta and gamma rays is in their different abilities to penetrate the irradiated material. Gamma 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 dosage can be applied within seconds, whereas several hours are required in the gamma radiation plant. (Fig. 1). [1]–[12],[21]

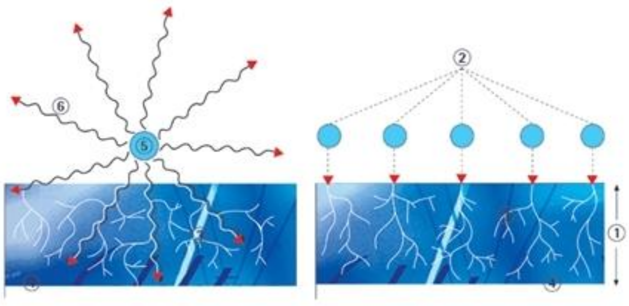

Fig. 1 Design of gamma rays (a) and electron rays (b), 1 – Penetration depth of an electron, 2 – Primary electron, 3 – Secondary electron, 4 – Irradiated material, 5 – Encapsulated Co – 60 Radiation source, 6 – Gamma rays [2]

Beta and gamma rays can be used for the irradiation of polyolefines, polyesters, halogen polymers and polyamides from the thermoplastics group, elastomers and thermoplastic

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elastomers. Some of them need the addition of a cross-linking agent. [1,6,7,8]

Radiation cross-linking usually improves strength, reduces creep, contributes to chemical resistance improvement and in many cases improves tribological properties. Effect of radiation cross-linking significantly improves temperature stability. Because of that, materials which belong to group of standard polymers can be used in applications, which would be in term of temperature stability intended only to constructive thermoplastic polymers.

II. EXPERIMENTAL

For this experiment polymethyl methacrylate (PMMA) PLEXIGLAS 8N; Evonik Industries AG, Germany was used. The prepared specimens were irradiated with doses of 66 and 99 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany [1-4].

The samples were made using the injection molding technology on the injection moulding machine ArburgAllrounder 420C. Processing temperature 220–260 °C, mold temperature 60 °C, injection pressure 80 MPa, injection rate 45 mm/s.

Instrumented nanohardness tests were done using a Nanoindentation Tester (NHT2) – Opx/Cpx, CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1. Load and unload speed was 100 mN/min. After a holding time of 90 s at maximum load 50 mN the specimens were unloaded. The indentation hardness HIT was calculated as maximum load to the projected area of the hardness impression according to:

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

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.

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

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

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. [8] [12] [41].

Determination of indentation creep C_{IT}:

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

Where h_1 is representing 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 (Fig. 2) [1] [7] [15].

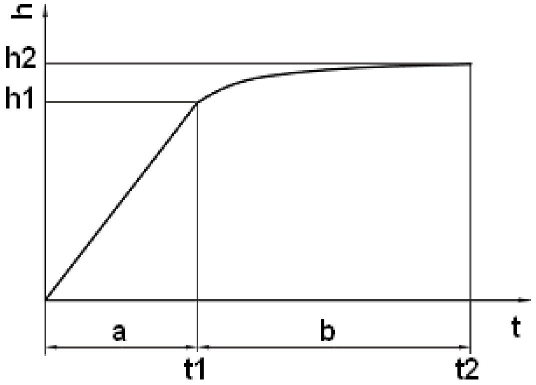

Fig. 2 Illustration of indentation creep parameters

Elastic part of the indentation work η_{IT} (Fig. 3):

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

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

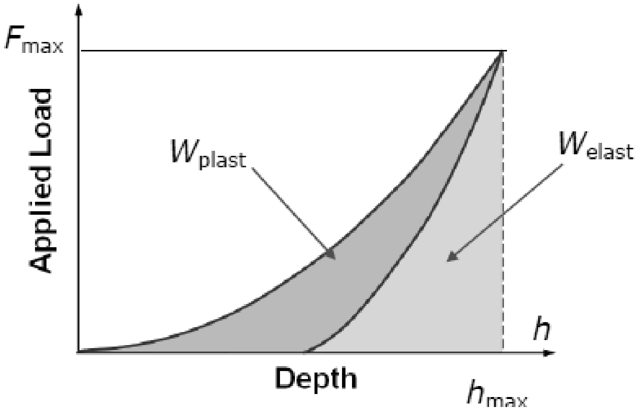

Fig. 3 Illustration of coefficient of back deformation

III. RESULTS AND DISCUSSION

For Instrumented nanohardness teset was used three different loads

A. Indentation load 10 mN

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found for non-irradiated PMMA. On the contrary, the highest values of indentation hardness were obtained for PMMA irradiated with dose of 66 kGy (by 11% lower in comparison with non irradiated PMMA), as can be seen at Fig. 4.

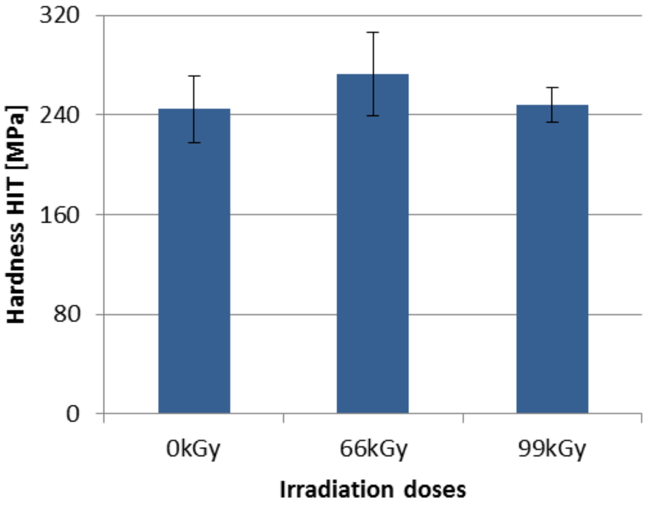

Fig. 4 Hardness HIT of PMMA vs. irradiation doses

Higher radiations influence significantly the nanohardness value. An indentation hardness decrease of the surface layer is caused by degradation of the tested specimen.

According to the results of measurements of nanohardness, it was found that the highest values of indentation modulus of elasticity were achieved at the PMMA irradiated with dose of 66 kGy (by 15% higher than compared with non-irradiated PMMA). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PMMA as is seen at Fig. 5.

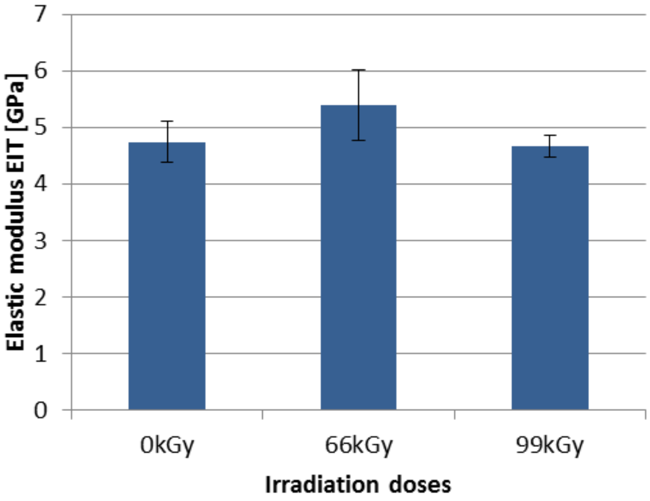

Fig. 5 Elastic modulus EIT of PMMA vs. irradiation doses

The lowest values of hardness Vickers were found for non irradiated PMMA. On the contrary, the highest values of hardness Vickers were obtained for PMMA irradiated with dose of 66 kGy (by 11% higher in comparison with non irradiated PMMA), as can be seen at Fig. 6.

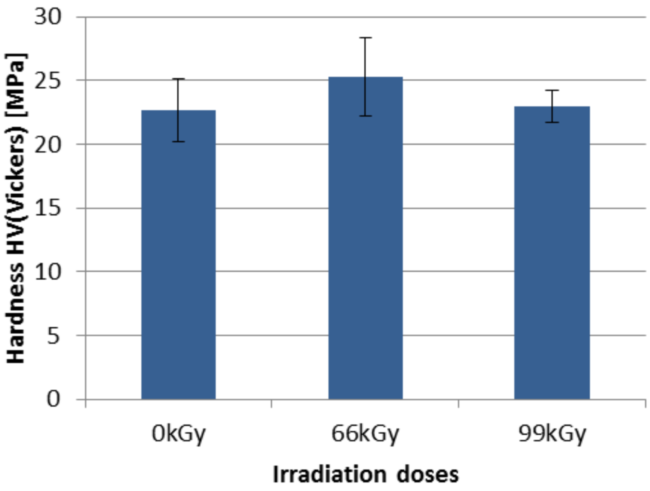

Fig. 6 Hardness Vickers of PMMA vs. irradiation doses

Other important material parameters obtained during the nanohardness test were elastic and plastic deformation work. The elastic deformation work We determines the reaction of material to applied (multiaxial) load with reversible deformation. The plastic part of the deformation work Wpl defines toughness of the tested material (surface layer) and its resistance to plastic deformation.

The greatest values of plastic deformation work were obtained for non-irradiated PMMA. The greatest values of elastic deformation work were obtained for PMMA irradiated with dose of 99 kGy. The lowest values of plastic and elastic deformation work were obtained for the PMMA irradiated with dose of 66 kGy. Radiation of specimens caused lower values of elastic as well as plastic deformation work which is apparent in Fig. 7.

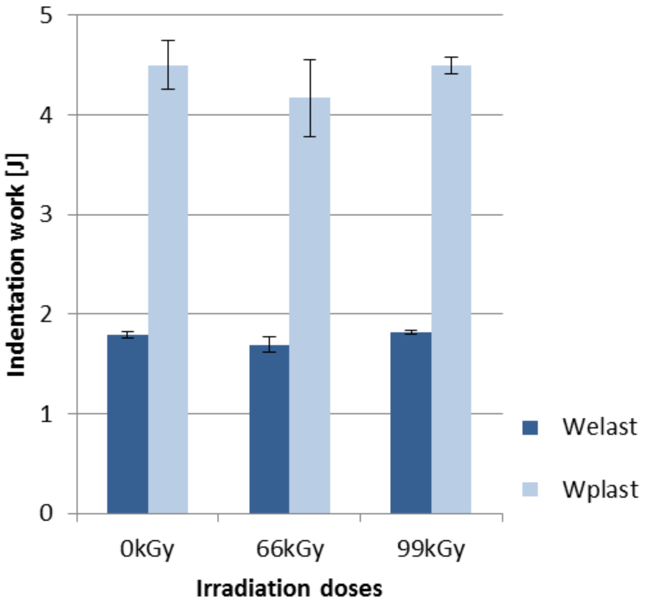

Fig. 7 Elastic and plastic deformation work of PMMA vs. irradiation doses

The greatest values of indentation creep were obtained for PMMA irradiated with dose of 99 kGy (by 5% higher in comparison with PMMA irradiated with dose of 66 kGy). The lowest values of indentation creep were obtained for PMMA irradiated with dose of 66 kGy.

Radiation of specimens caused decrease of indentation creep and subsequent little increase of indentation creep which is apparent in Fig. 8.

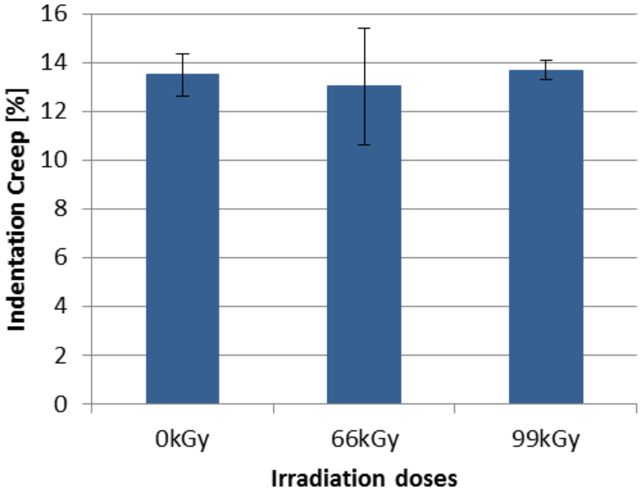

Fig. 8 Indentation Creep of PMMA vs. irradiation doses

The lowest values of back deformation coefficient n_{IT} were found for the PMMA irradiated with dose of 30 kGy. On the contrary, the highest values of back deformation coefficient n_{IT} were obtained for non-irradiated PMMA (by 12% higher in comparison with the PMMA irradiated with dose of 30 kGy), as can be seen at Fig. 9.

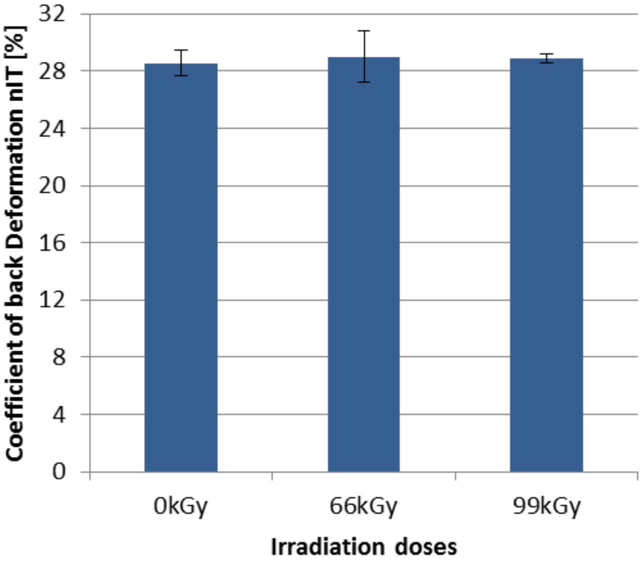

Fig. 9 Coefficient of back deformation n_{IT} vs. irradiation doses

B. *Indentation load 50 mN*

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found for the PMMA irradiated by a dose of 99 kGy. On the contrary, the highest values of indentation hardness were obtained for non-irradiated PMMA (by 5% higher in comparison with PMMA irradiated by a dose of 99 kGy), as can be seen at Fig. 10.

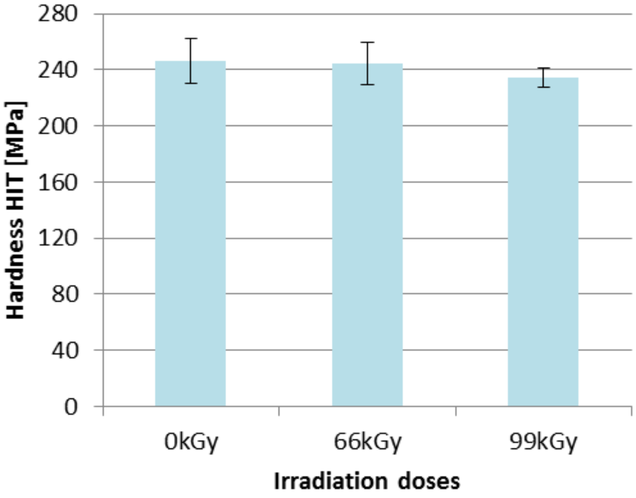

Fig. 10 Hardness HIT of PMMA vs. irradiation doses

Higher radiation dose does not influence significantly the nanohardness value. An indentation hardness decrease of the surface layer is caused by degradation of the tested specimen.

According to the results of measurements of nanohardness, it was found that the highest values of indentation modulus of elasticity were achieved at the non-irradiated PMMA (by 5% higher than compared with PMMA irradiated with dose of 99 kGy). On the contrary, the lowest values of the indentation modulus of elasticity were found for PMMA irradiated with dose of 99 kGy as is seen at Fig 11.

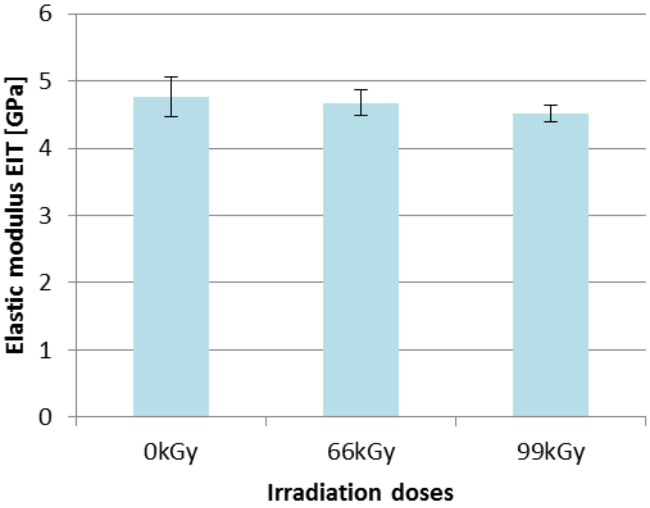

Fig. 11 Elastic modulus EIT of PMMA vs. irradiation doses

The lowest values of hardness Vickers were found for PMMA irradiated with dose of 99 kGy. On the contrary, the highest values of hardness Vickers were obtained for non-irradiated PMMA (by 5% higher in comparison with PMMA irradiated with dose of 99 kGy), as can be seen at Fig. 12.

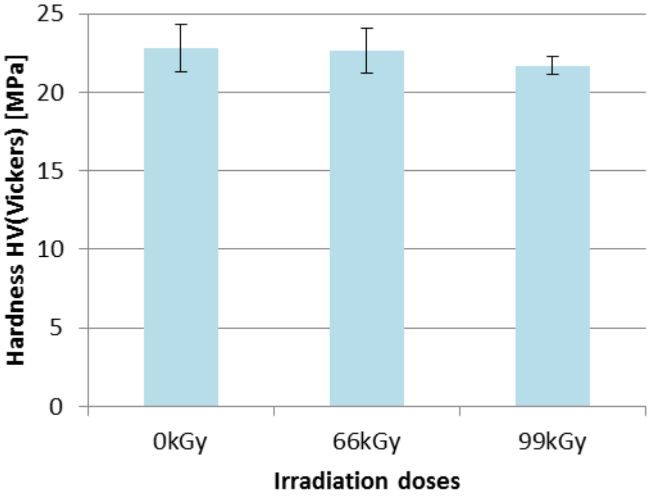

Fig. 12 Hardness Vickers of PMMA vs. irradiation doses

The greatest values of plastic and elastic deformation work were obtained for PMMA irradiated with dose of 99 kGy. The lowest values of elastic deformation work were obtained for non-irradiated PMMA. The lowest values of plastic deformation work were obtained for PMMA irradiated with dose of 66 kGy. Radiation of specimens caused higher values of elastic as well as plastic deformation work which is apparent in Fig.13.

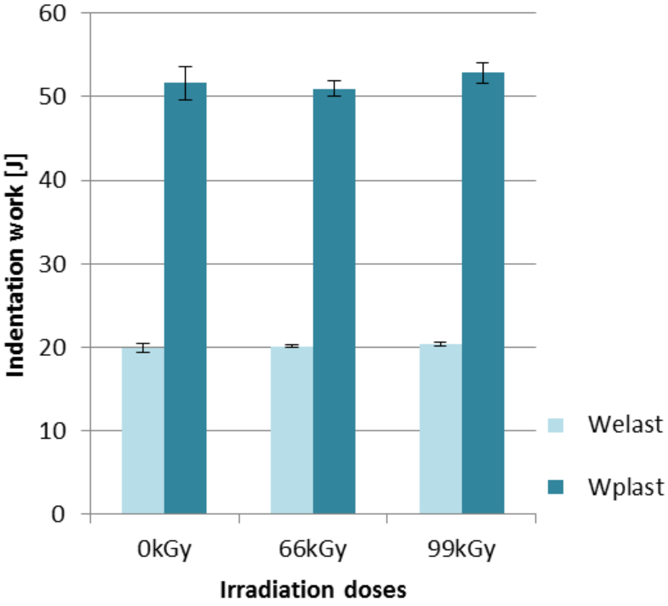

Fig. 13 Elastic and plastic deformation work of PMMA vs. irradiation doses

The greatest values of indentation creep were obtained for PMMA irradiated with dose of 99 kGy. The lowest values of indentation creep were obtained for PMMA irradiated with dose of 66 kGy.

Radiation of specimens caused decrease of indentation creep and subsequent little increase of indentation creep which is apparent in Fig. 14.

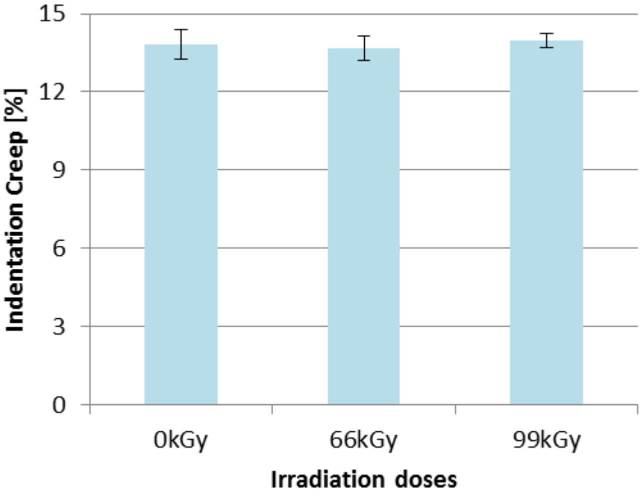

Fig. 14 Indentation Creep of PMMA vs. irradiation doses

The lowest values of back deformation coefficient n_{IT} were found for PMMA irradiated by a dose of 99 kGy. On the contrary, the highest values of back deformation coefficient n_{IT} were obtained for PMMA irradiated by a dose of 66 kGy (by 2% higher in comparison with the PMMA irradiated by a dose of 99 kGy), as can be seen at Fig. 15.

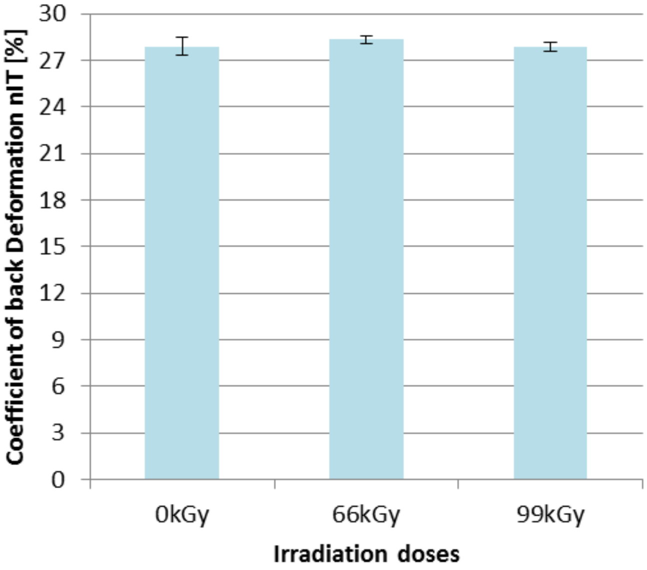

Fig. 15 Coefficient of back deformation n_{IT} vs. irradiation doses

C. Indentation load 250 mN

The values measured during the nanohardness test showed that the lowest values of indentation hardness were found for non-irradiated PMMA. On the contrary, the highest values of indentation hardness were obtained for PMMA irradiated by a dose of 99 kGy (by 4% higher in comparison with non-irradiated PMMA), as can be seen at Fig.16.

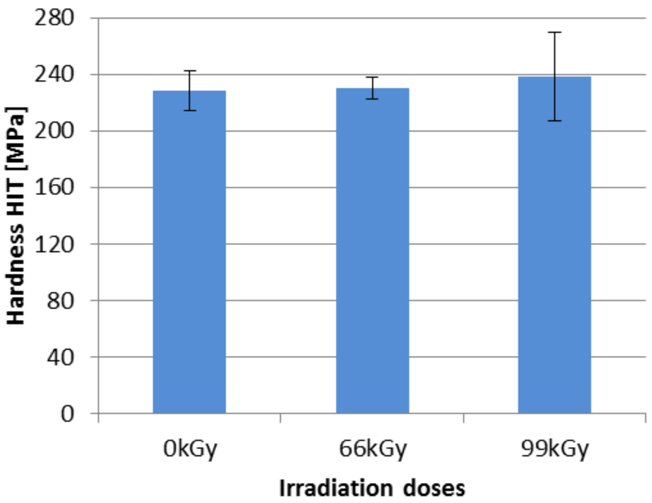

Fig. 16 Hardness HIT of PMMA vs. irradiation doses

Higher radiation dose does not influence significantly the nanohardness value. An indentation hardness decrease of the surface layer is caused by degradation of the tested specimen.

According to the results of measurements of nanohardness, it was found that the highest values of indentation modulus of elasticity were achieved at PMMA irradiated with dose of 99 kGy (by 1% higher than compared with non-irradiated PMMA). On the contrary, the lowest values of the indentation modulus of elasticity were found for non-irradiated PMMA as is seen at Fig 17.

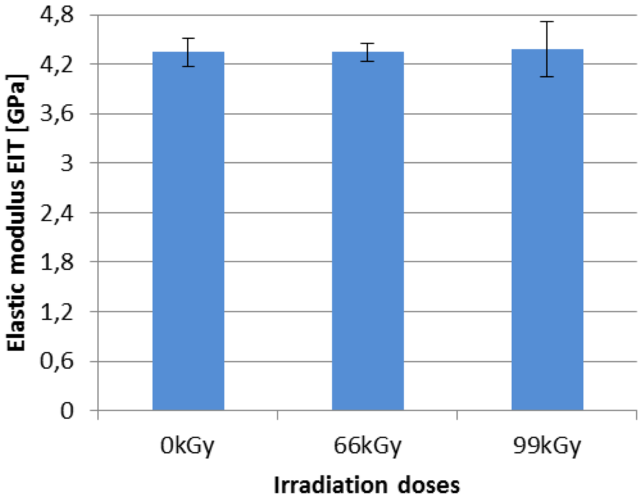

Fig. 17 Elastic modulus EIT of PMMA vs. irradiation doses

The lowest values of hardness Vickers were found for non-irradiated PMMA. On the contrary, the highest values of hardness Vickers were obtained for PMMA irradiated with dose of 99 kGy (by 4% higher in comparison with non-irradiated PMMA), as can be seen at Fig. 18.

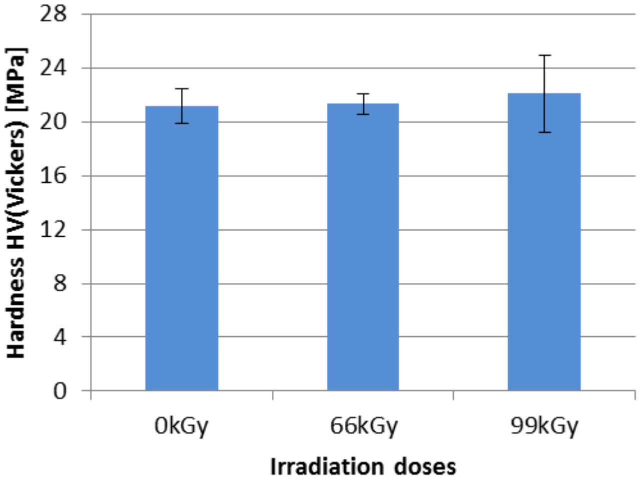

Fig. 18 Hardness Vickers of PMMA vs. irradiation doses

The greatest values of elastic deformation work were obtained for PMMA irradiated with dose of 99 kGy. The greatest values of plastic deformation work were obtained for non-irradiated PMMA. The lowest values of elastic and plastic deformation work were obtained for PMMA irradiated with dose of 66 kGy. Radiation of specimens not caused significantly changes of values of plastic and elastic deformation work which is apparent in Fig. 19.

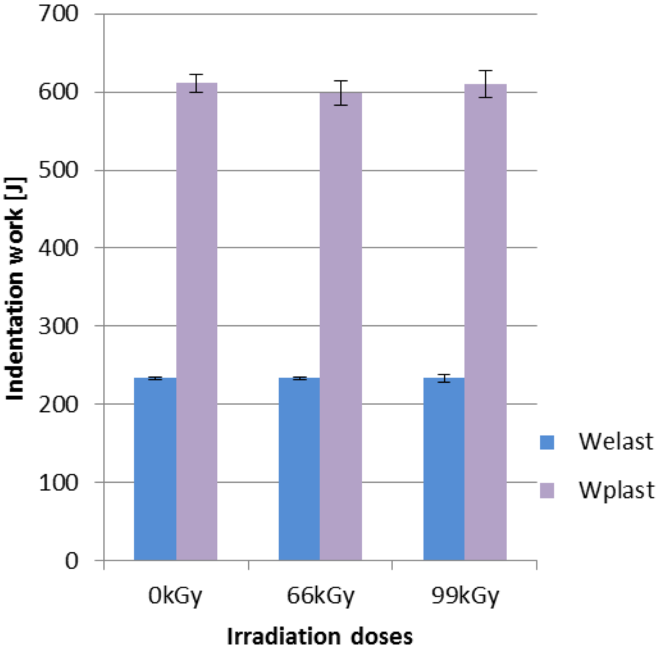

Fig. 19 Elastic and plastic deformation work of PMMA vs. irradiation doses

The greatest values of indentation creep were obtained for PMMA irradiated with dose of 99 kGy. The lowest values of indentation creep were obtained for PMMA irradiated with dose of 66 kGy.

Radiation of specimens caused decrease of indentation creep and subsequent increase of indentation creep which is apparent in Fig. 20.

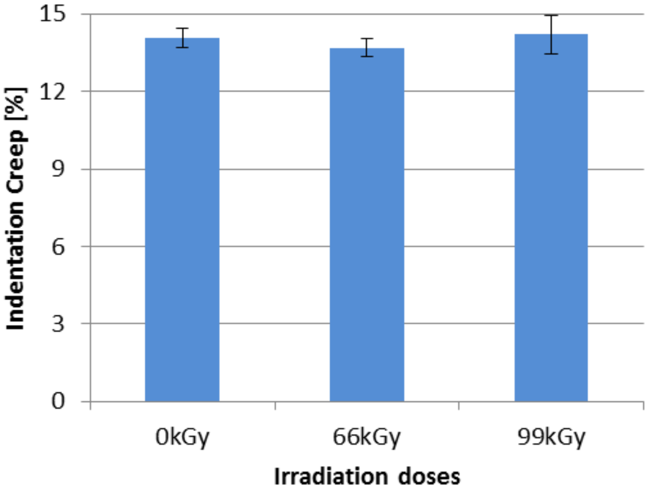

Fig. 20 Indentation Creep of PMMA vs. irradiation doses

The lowest values of back deformation coefficient n_{IT} were found for the non-irradiated PMMA. On the contrary, the highest values of back deformation coefficient n_{IT} were obtained for PMMA irradiated by a dose of 99 kGy (by 2% higher in comparison with the non-irradiated PMMA), as can be seen at Fig. 21.

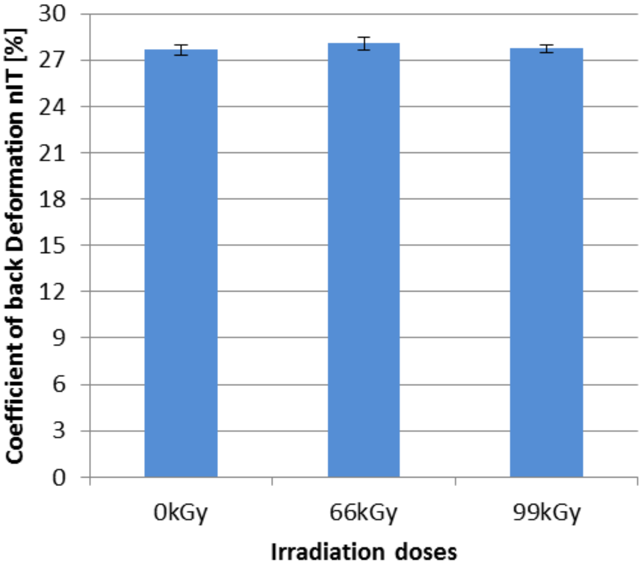

Fig. 21 Coefficient of back deformation n_{IT} vs. irradiation doses

IV. CONCLUSION

For measurement with load of 10mN we obtained lowest indentation depth for non-irradiated PMMA. The greatest values were obtained for PMMA irradiated with dose of 99 kGy, as can be seen at Fig. 22.

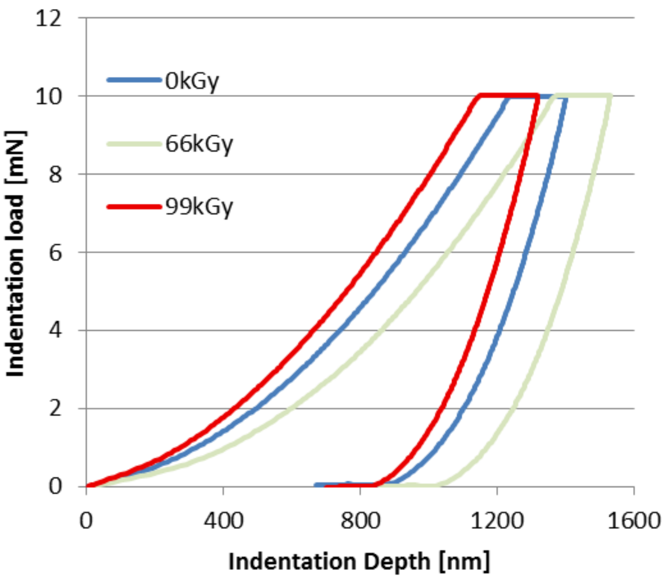

Fig. 22 Indentation load vs. Indentation depth

The correlation between force and depth of indentation into surface layer is apparent in Fig. 23. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens. The PMMA irradiated with dose of 66 kGy showed slightly lower hardness as well as increasing impression of the indenter in the surface layer.

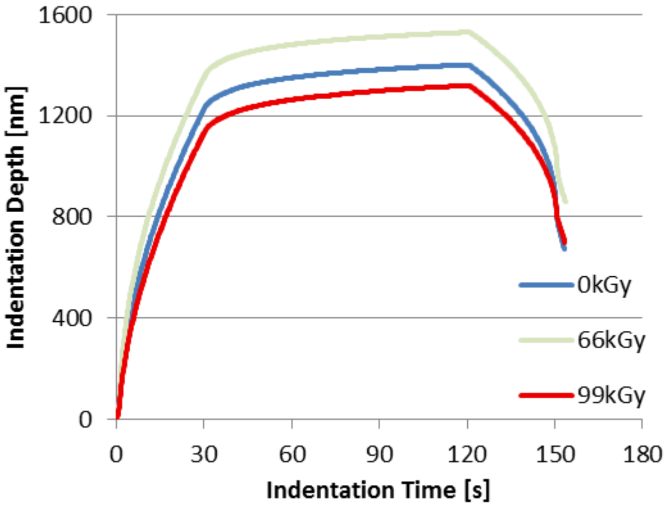

Fig. 23 Indentation depth vs. Indentation time

For measurement with load of 50mN we obtained lowest indentation depth for PMMA irradiated with dose of 99 kGy.

The greatest values were obtained for PMMA irradiated with dose of 66 kGy, as can be seen at Fig. 24.

The correlation between force and depth of indentation into surface layer is apparent in Fig. 25. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens.

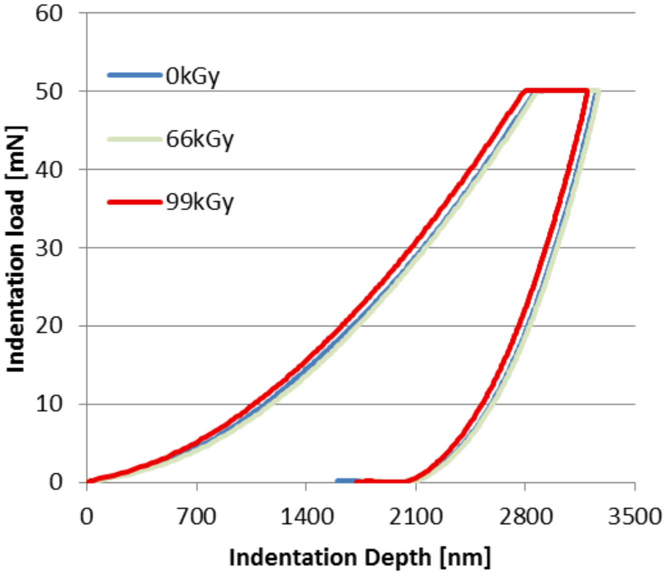

Fig. 24 Indentation load vs. Indentation depth

The PMMA irradiated with dose of 66 kGy showed slightly lower hardness as well as increasing impression of the indenter in the surface layer.

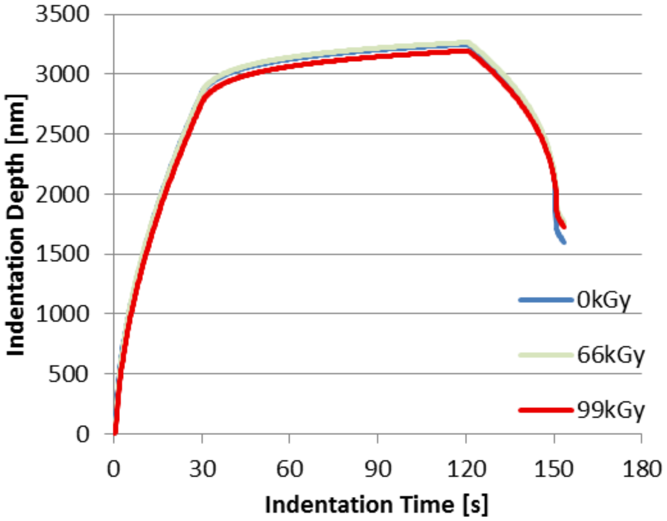

Fig. 25 Indentation depth vs. Indentation time

For measurement with load of 250mN we obtained lowest indentation depth for PMMA irradiated with dose of 99 kGy. The greatest values were obtained for non-irradiated PMMA, as can be seen at Fig. 26.

The correlation between force and depth of indentation into surface layer is apparent in Fig. 27. It demonstrated the influence of radiation on the change of mechanical properties in the surface layer of specimens. The PMMA irradiated with dose of 66 kGy showed slightly lower hardness as well as increasing impression of the indenter in the surface layer.

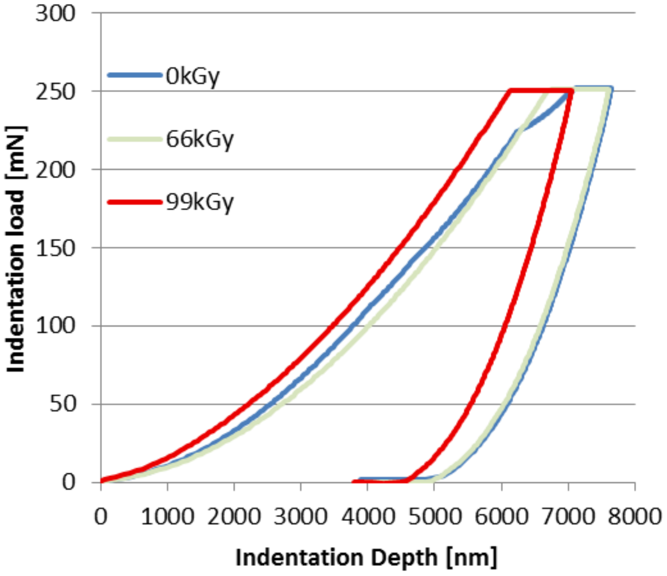

Fig. 26 Indentation load vs. Indentation depth

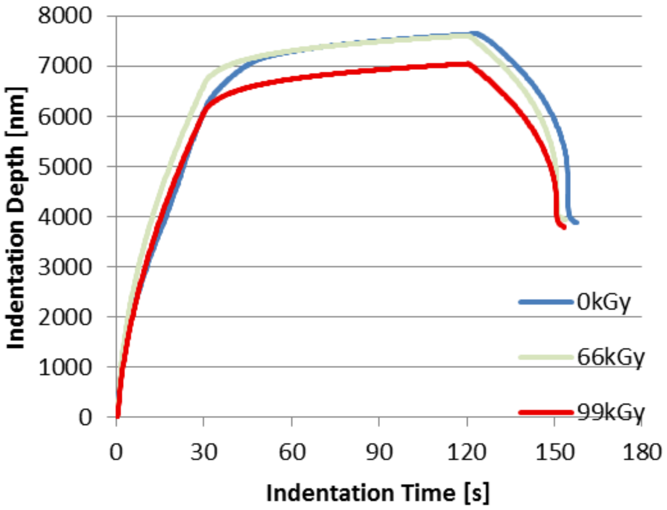

Fig. 27 Indentation depth vs. Indentation time

The properties of surface layer of Polymethyl methacrylate modified by beta radiation reported very little improvement. The nanohardness values increased about 10% at higher load as 10mN but only about 4% with indentation load 250 mN. Stiffness of surface layer decreased slowly by 5% as a result of radiation. Changes of behavior in the surface layer were confirmed by final values of plastic deformation work whose values decreased in correlation with the increasing radiation dose. Elastic deformation was slowly increasing with radiation dose. The highest values of micromechanical properties were reached at non-irradiatid PMMA. The results of nanomechanical properties of surface layer of modified polymethyl methacrylate show very small increasing of end-user properties. The improvements of PMMA after irradiation were on the border of distinguishability due to variance of measured data.

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.1.05/2.1.00/03.0089.

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