

# Properties of Irradiated PA11 by Accelerated Electrons

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**Abstract**—Radiation processing involves the use of natural or manmade sources of high energy radiation on an industrial scale. The principle of radiation processing is the ability of high energy radiation to produce reactive cations, anions and free radicals in materials. The industrial applications of the radiation processing of plastics and composites include polymerization, cross-linking, degradation and grafting. Radiation processing mainly involves the use of either electron beams from electron accelerators or gamma radiation from Cobalt-60 sources. The PA11 polyamide 11 tested showed significant changes of temperature stability and mechanical properties after irradiation. From this point view, new applications could also be seen in areas with service temperatures higher than their former melting point. The comparison of the temperature stability and mechanical properties of irradiated and non irradiated PA11 is presented in this paper.

**Keywords**—Polyamide 11, irradiation, mechanical properties

## I. INTRODUCTION

**P**OLYMERS 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 properties

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. Radiation processing with an electron beam offers several distinct advantages when compared with other radiation sources, particularly  $\gamma$ -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  $\gamma$ -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. These are not only capable of converting monomeric and oligomeric liquids, but also can produce, due to cross-linking, major changes in the properties of solid polymers. The cross-linking level can be adjusted by the irradiation dosage. The absorbed dosage means the value of energy of ionizing radiation absorbed by a unit of mass of the processed material. The unit of absorbed dose is 1 Gray (1 Gy = 1J/kg). 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, 2]

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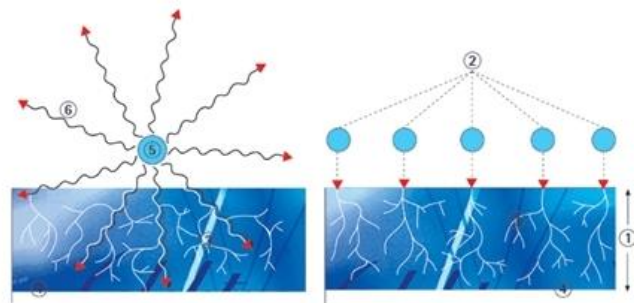


Fig. 1 Design of Gamma Rays (a) and Electron Rays (b), 1 – Penetration depth of an electron, 2 – Primary electron, 3 – Secondary electron, 4 – Irradiated material, 5 – Encapsulated Co – 60 Radiation source, 6 – Gamma Rays [2]

Beta and gamma rays can be used for the irradiation of polyolefines, polyesters, halogen polymers and polyamides from the thermoplastics group, elastomers and thermoplastic elastomers. Some of them need the addition of a cross-linking agent. Polymers can be classified into two groups according to their response to ionizing radiation. One group exhibits predominant cross-linking, the other predominant chain scission. [1, 2]

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. MATERIAL AND METHODS

### A. Material Preparation

As the basic polymer material was used Polyamide 11 (V-PTS-CREAMID-11T\*M600/13transparent) produced in PTS Plastic-Technologie-Service. An ARBURG Allrounder 420C Advance Injection molding machine was used for sample preparation, with the processing conditions to comply with the PA11 producer's recommendations. Irradiation of tested PA11 polymer was performed with the kind help of BGS Germany, in the BGS Wiehl plant using accelerated electrons with a dosage range of 0 to 198kGy. The mechanical properties and the temperature stability of non-irradiated and irradiated PA11 were tested after irradiation.

### B. Used Methods for the Testing

Firstly, the mechanical properties were measured. Tensile test was carried out on tensile machine ZWICK 1456, according to standard CSN EN ISO 527-1, 527-2. Used rate: 50mm/min. Test data was processed by Test Expert Standard software and modulus (E [MPa]) and tensile stress ( $\sigma_t$  [MPa]) were determined. The hardness was measured using a HPE – D Berreiss hardness tester type, and the Shore D Method. The shape and the dimensions of the testing samples were in accord with the CSN 621431 standard. Stress corrosion cracking test was measured according to standard ISO 22 088-3. Lastly, were measured the thermo-mechanical properties and the temperature stability. Perkin – Elmer Thermal Analyser TMA7 was used for the thermo-mechanical analysis; heated from 50°C to 400°C at 20°C/min, hold for 1 min at 50°C. The temperature stability was determined by visual observation in the temperature chamber.

## III. RESULTS AND DISCUSSION

The mechanical and temperature behaviour of polyamide 11 (PA11), before and after irradiation, was studied. For easier of evaluation of the measured data, and the comparison of the irradiated polymer with a non-irradiated one, both real measured data and changes of measured properties were used

in graphs. The property of the non-irradiated polymer had the value of 100%, while others were expressed in % as the ratio of measured property of irradiated polymers to the same property of non-irradiated polymer.

### A. Tensile Test, Hardness and Corrosion under stress

Irradiation affects the mechanical properties of the PA11 studied at the ambient temperature for hardness and at 23°C and 80°C for tensile test. Corrosion under stress was studied also at ambient temperature.

#### 1) Tensile Strength and E-modulus

Irradiation affects the tensile strength of the PA11 studied at the ambient temperature (Fig. 2).

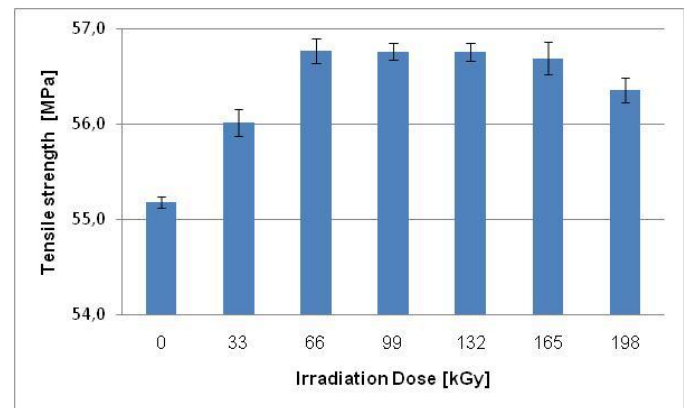


Fig. 2 PA11 Tensile Strength at the ambient temperature

At the ambient temperature tensile strength of irradiation PA11 with a dosage of 66kGy, was increased about 1,2 MPa compared to non-irradiated PA11.

Irradiation affects the tensile strength of the PA11 studied at 80°C (Fig. 3).

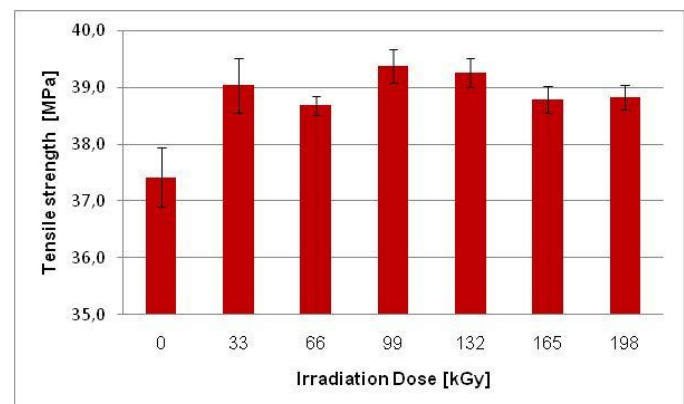


Fig. 3 PA11 Tensile Strength at 80°C

At 80°C tensile strength of irradiation PA11 with a dosage of 99kGy was increased about 1,9 MPa compared to non-irradiated PA11.

In the Fig. 4 it is possible to see comparison of PA11 tensile strength in percentage at the ambient temperature and 80°C.

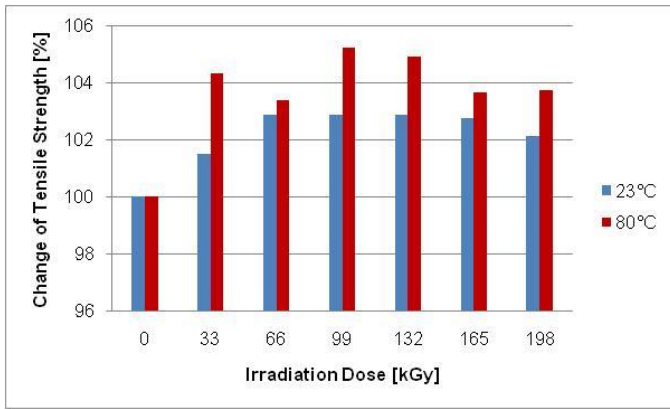


Fig. 4 PA11 Tensile Strength at 23°C and 80°C

The tensile strength rises only minimally, about 3% after irradiation with a dosage of 66 kGy at 23°C but at 80°C the tensile strength rises by 5% after irradiation with a dosage of 99 kGy. From the point-of-view of its tensile strength a dosage of irradiation of 99 kGy would seem to be optimal (Fig. 4).

Irradiation affects the E-modulus of the PA11 studied at both the ambient (Fig. 5) and increased temperature.

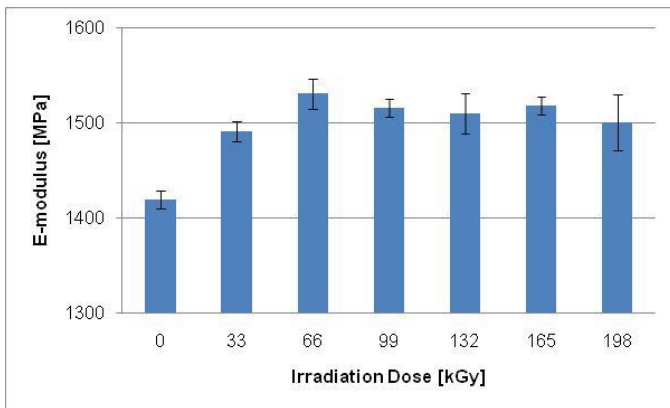


Fig. 5 PA11 E-modulus at the ambient temperature

At the ambient temperature PA11 E-modulus is increased up to 66kGy and then it is begun to decrease with increased irradiation dose.

Irradiation affects the E-modulus of the PA11 studied at 80°C (Fig. 6). PA11 E-modulus at 80°C is increased up to 33kGy and then it is begun to decrease with irradiated dose up to 165kGy then there is increasing about 70MPa at 198kGy.

In the Fig. 7 it is possible to see comparison of PA11 E-modulus in percentage at the ambient temperature and 80°C.

PA11 E-modulus at 23°C is increased by 8% for irradiated PA11 with a dosage of 66kGy. PA11 E-modulus at 80°C is increased by 7% for irradiated PA11 with a dosage of 198kGy.

From the point-of-view of its elastic modulus a dosage of irradiation of 198 kGy would seem to be optimal (Fig. 7).

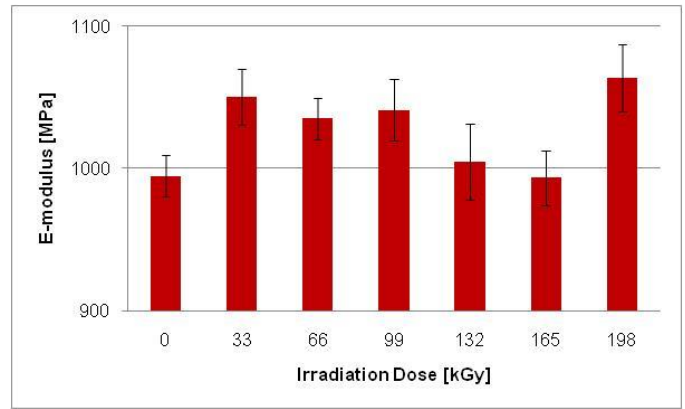


Fig. 6 PA11 E-modulus at 80°C

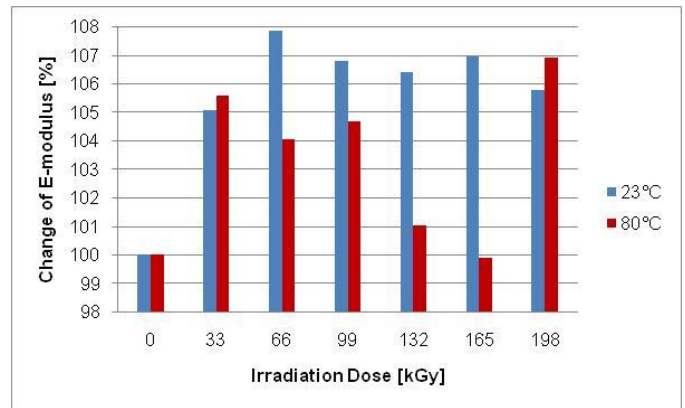


Fig. 7 PA11 E-modulus at 23°C and 80°C

## 2) Hardness

Irradiation affects the hardness of the PA11 studied at the ambient temperature (Fig. 8).

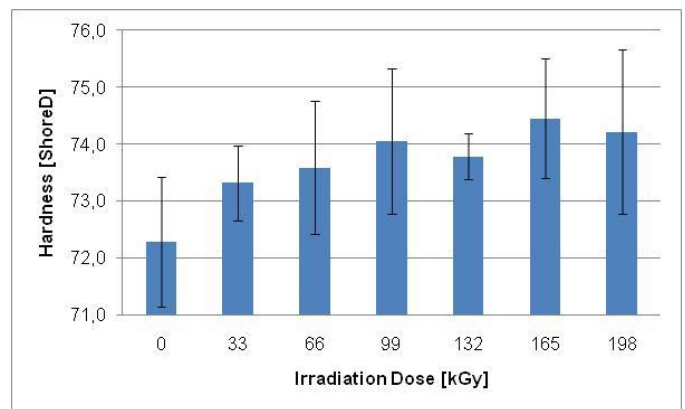


Fig. 8 PA11 Hardness at the ambient temperature

At the ambient temperature PA11 Shore D hardness is slightly increased with the increase irradiation dose. The Shore D hardness of irradiated PA11 is increased by 2 Shore D compared to non-irradiated PA11 at 165kGy.

In the Fig. 9 it is possible to see comparison of PA11 Shore D hardness in percentage at the ambient temperature.

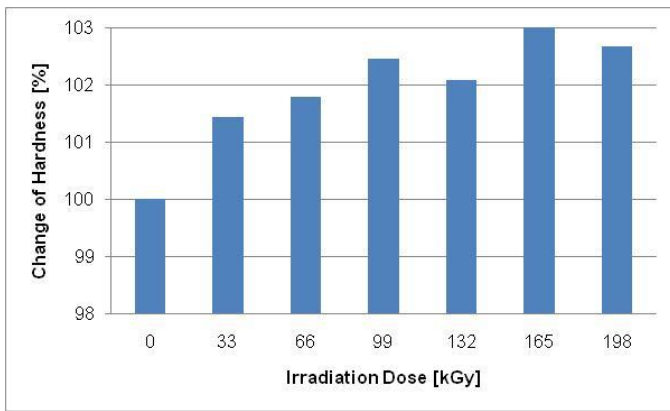


Fig. 9 Comparison of PA11 Hardness

No significant changes of hardness were found after the irradiation on the PA11 sample. A maximum increase of hardness (i.e. about 3%) was measured after the irradiation dosage of 165kGy. Higher doses of irradiation had no significant effect on the Shore D Hardness. From the point-of-view of its Shore D hardness a dosage of irradiation of 165 kGy would seem to be optimal (Fig. 9).

### 3) Stress corrosion cracking

For the measurement of stress corrosion cracking was used standard ISO 22 088-3. The measurement was performed at constant deformation; the radius of bending was 94 mm. The measurement of specimens was performed regularly every 24 hours.

Non-irradiated and irradiated PA11 specimens were examined in the reference environment and then in the ethyleneglykol environment (Fig. 10 – 18).

PA11 specimens were not destructed (any cracks) in the reference environment for long time of measurement.

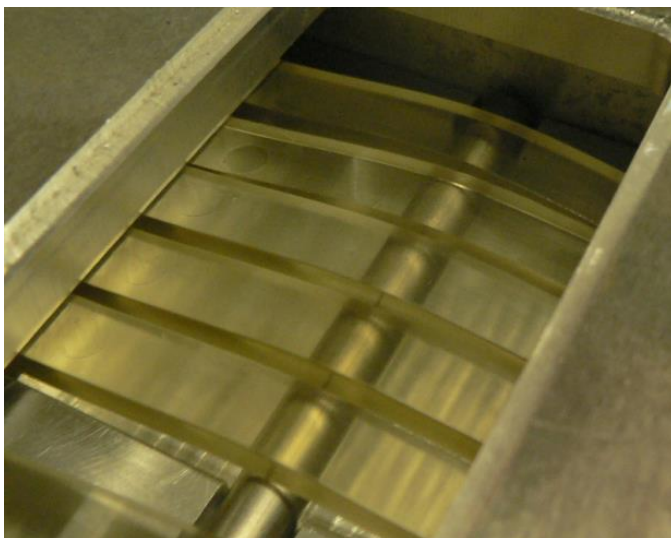


Fig. 10 Non-irradiated PA11 specimens placed in the reference environment

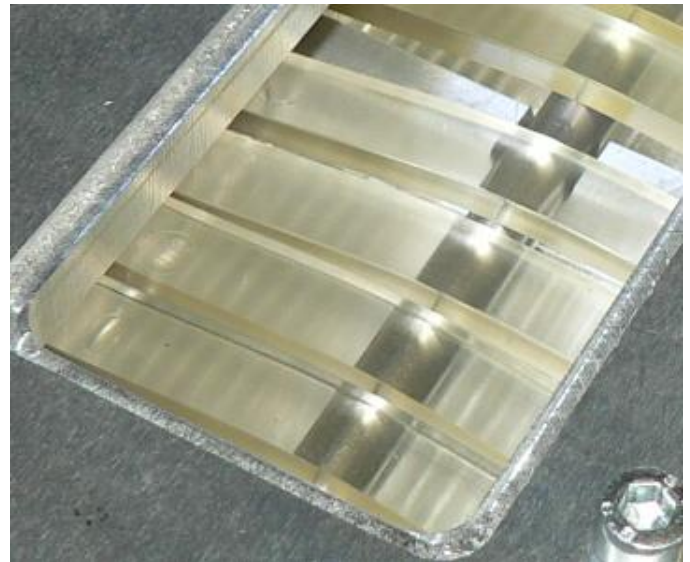


Fig. 11 Irradiated PA11 specimens placed in the reference environment

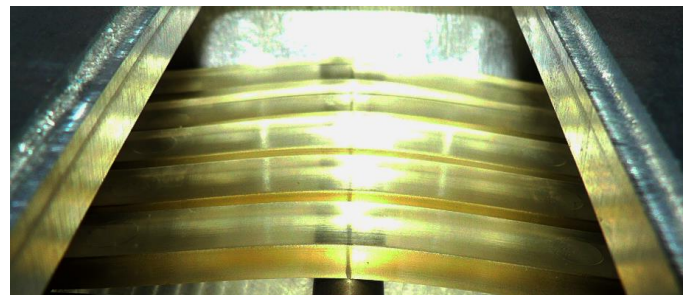


Fig. 12 Non-irradiated PA11 specimens placed in the reference environment after 168 hours

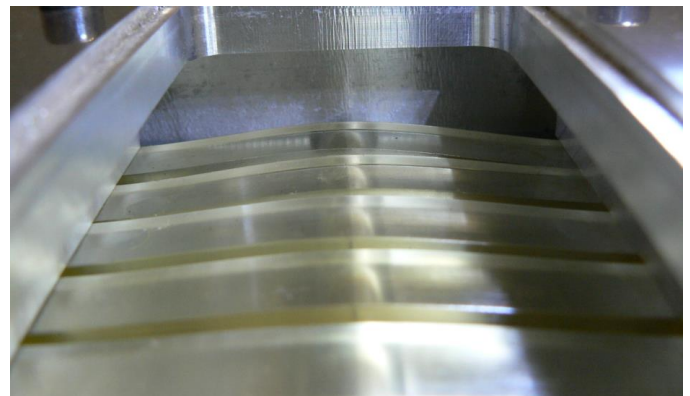


Fig. 13 Irradiated PA11 specimens placed in the reference environment after 168 hours

Irradiated PA11 specimens started cracking in the ethyleneglykol environment after 24 hours, more visible cracks were seen after 72 hours. Non-irradiated PA11 specimens started cracking in the ethyleneglykol environment after 72 hours and then these cracks were not on the increase with time. Stress corrosion cracking test was completed after 168 hours.



Fig. 14 Non-irradiated PA11 specimens placed in the ethyleneglykol environment after 24 hours



Fig. 17 Irradiated PA11 specimens placed in the ethyleneglykol environment after 72 hours

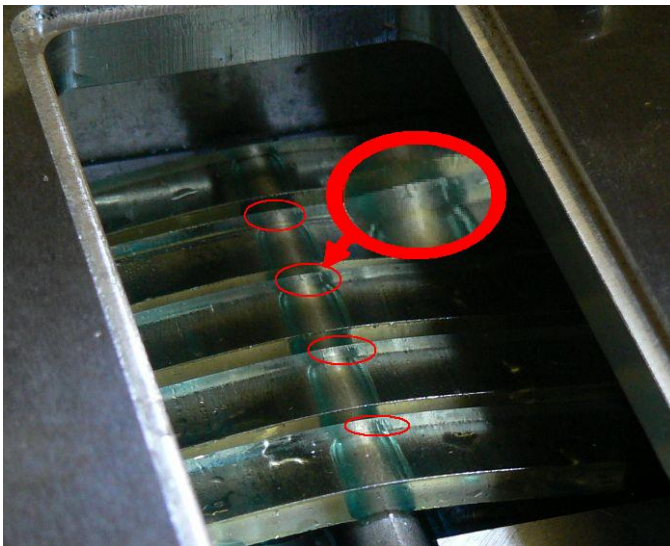


Fig. 15 Irradiated PA11 specimens placed in the ethyleneglykol environment after 24 hours

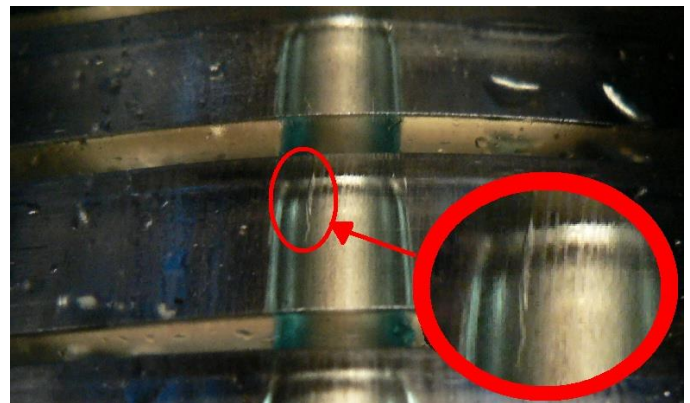


Fig. 18 Irradiated PA11 specimens placed in the ethyleneglykol environment after 120 hours

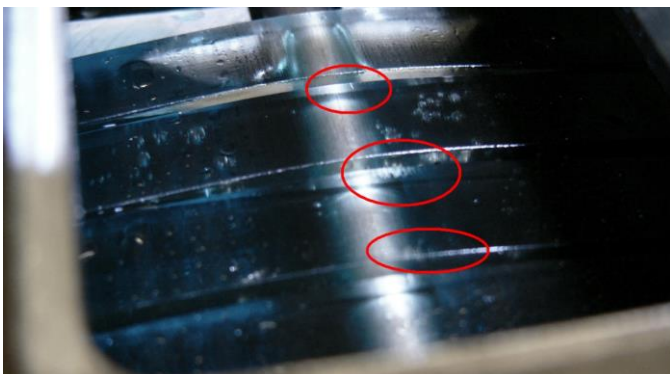


Fig. 16 Non-irradiated PA11 specimens placed in the ethyleneglykol environment after 72 hours

### B. Temperature Stability

The temperature stability of PA11 was measured. The temperature stability was evaluated by TMA measurement and by visual observation. The graphical depiction of TMA results and description of a test record of the temperature stability inside a temperature chamber are numerically and colorfully distinguished according to the dose of radiation. Specimens are numbered from 1 to 7 according to dose of irradiation (number 1 means non-irradiated polymer – 0kGy, number 7 the specimen with the highest dose – 198kGy).

#### 1) Thermo-mechanical analysis

Irradiation affects the thermo-mechanical properties of the studied PA11 (Fig. 19).

Non-irradiated PA11 is melted at the temperature 200°C. Irradiated PA11 at 33kGy is melted at the temperature 330°C. PA11 with the increased dose above 66kGy have not lost the temperature stability up to 350°C (Fig. 19). Irradiated PA11 specimens above the dose of irradiation 66 kGy evince the significant improvement of temperature stability.

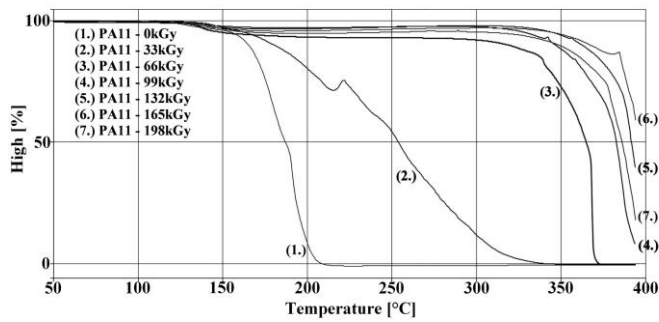


Fig. 19 Thermo-mechanical Analysis of PA11

2) Visual Observation of PA11 in the Temperature Chamber

The visual observation of sample behaviour at 100°C is given on Fig. 20. Specimens are fitted horizontally in the temperature chamber and loaded by the bending moment both from its own weight and the weight on the end of specimen. Specimens are not deformed. The surface quality and the colour of polymer is the same like before the exam.

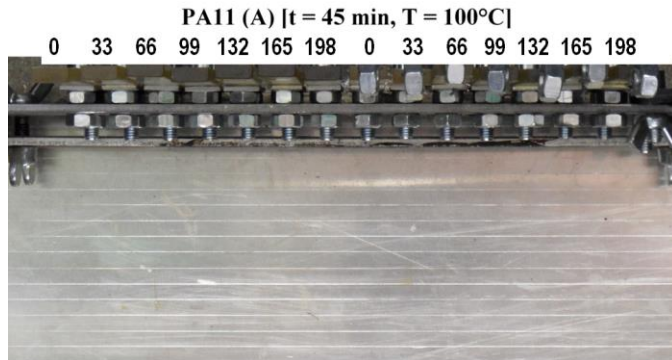


Fig. 20 PA11 specimen deformation at 100°C

The visual observation of sample behaviour at 150°C is given on Fig. 21.

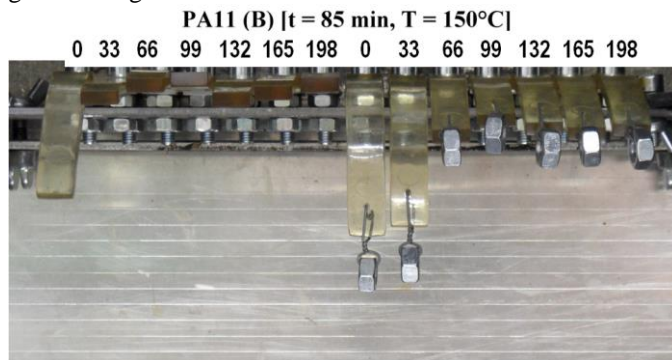


Fig. 21 PA11 specimen deformation at 150°C

The visual observation of sample behavior at 200°C is given on Fig. 22. At 200°C the specimen from non-irradiated PA11 is totally destroyed while the polymer irradiated by the dose of 198kGy keeps its cross-section without changes, there is only deformation by its own weight. The surface quality of polymer is the same like before the exam.

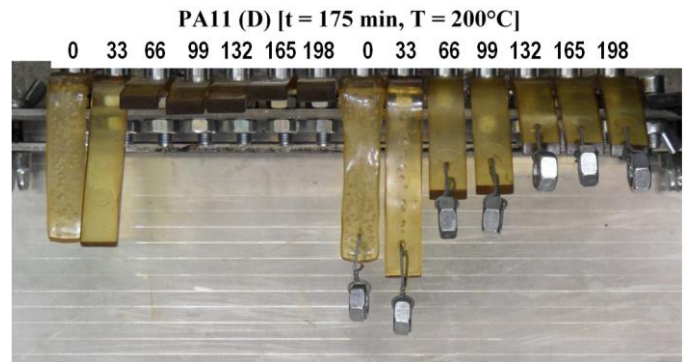


Fig. 22 PA11 specimen deformation at 200°C

The visual observation of sample behavior at 220°C is given on Fig. 23. At 220°C the non-irradiated PA11 specimen is totally melted while the polymer irradiated by the dose of 198kGy keeps its cross-section without changes, there is only deformation by its own weight. The surface quality of polymer is the same like before the exam.

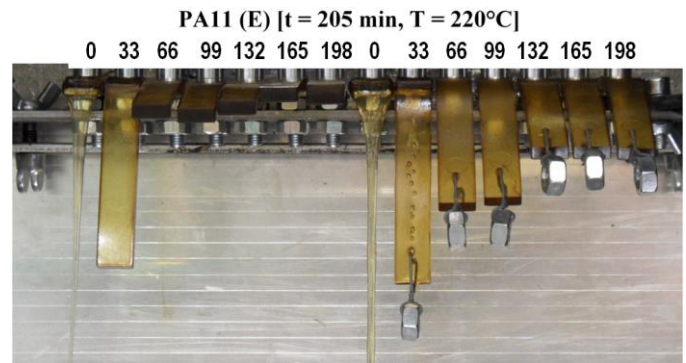


Fig. 23 PA11 specimen deformation at 220°C

The visual observation of sample behavior at 250°C is given on Fig. 24. The irradiated PA11 by the dose of 198kGy keeps its cross-section without changes; there is only deformation by its own weight. The surface quality and the colour of polymer start to deteriorate.

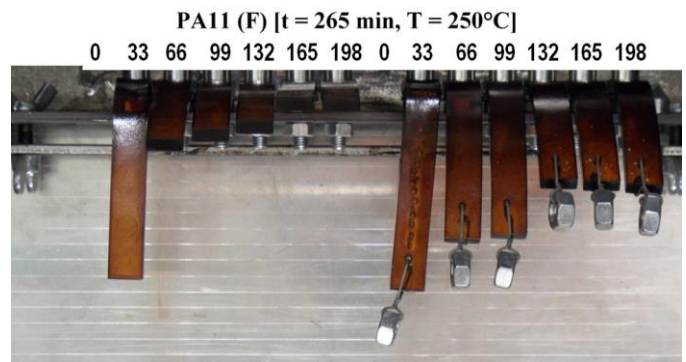


Fig. 24 PA11 specimen deformation at 250°C

The visual observation of sample behavior at 300°C is given on Fig. 25.

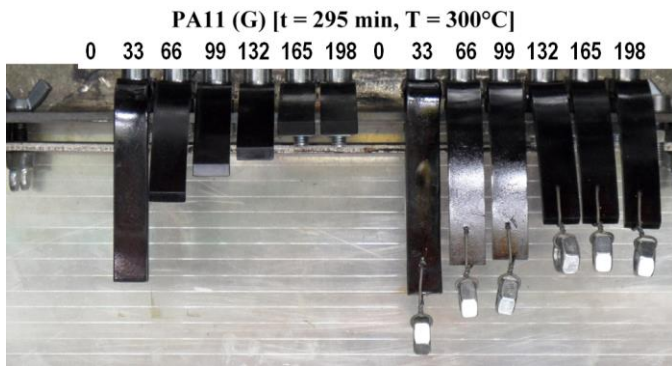


Fig. 25 PA11 specimen deformation at 300°C

Specimen deformation is decreasing with increasing dose of radiation at elevated temperature. At 300°C the specimen from not irradiated PA11 is totally melted while the polymer irradiated by the dose of 198kGy keeps its cross-section without changes, there is only deformation by its own weight. The surface quality worsens and the colour of polymer specimen change due to thermo-oxidation.

The temperature stability of polymers is very low in comparison with other construction materials. Experiments done in this project showed that irradiation cross-linking markedly affected the temperature stability of the PA11 studied. The higher the irradiation dosage, the better is the temperature stability of studied polymers. The tested specimen remained without dimensional changes at 150°C, after irradiation with a dosage of more than 66 kGy. The same specimen, at temperatures higher than 220°C, creates colour changes due to thermal oxidation - but its dimension/cross-section remains without change. Its better temperature stability makes possible to use this polymer - even at service temperatures higher than its former melting point.

#### IV. CONCLUSION

Plastics are by far the largest group of polymeric materials being processed by electron beam irradiation. Cross-linking of polyolefins, PVC, polyesters, polyurethanes, polyamides, fluoropolymers and fiber-reinforced composites are a common practice. Radiation cross-linking of polyamides requires considerably less overall energy and space, and is faster, more efficient and more environmentally acceptable. The disadvantage of electron beam cross-linking is a more or less nonuniform dose distribution. This can happen, particularly in thicker objects, due to intrinsic dose-depth profiles of electron beams. Another problem can be a nonuniformity of rotation of cylindrical objects as they traverse a scanned electron beam. However, the mechanical properties often depend on the mean cross-link density. [1]

As can be seen from the tests results, the irradiation cross-linking improves the PA11 mechanical properties. The improvement is more considerable in case of higher temperature (80 °C), as a consequence of creation of cross-link (during irradiation cross-linking) resulting in protraction of

macromolecular chain, which is thus more flexible during thermal load than individual shorter macromolecular chains.

Irradiation improves the thermal properties of polymer. Tested polymer (PA11) shows better temperature stability after irradiation. Irradiation significantly extends the application area of polymers. The service temperature can be higher than the melting point of not irradiated polymers.

The results of the measurements of PA11 after irradiation showed significant changes of its mechanical and thermo-mechanical properties. The tensile strength rises by 3%, after irradiation with a dosage of 66 kGy at the temperature 23°C and at the temperature 80°C the tensile strength rises by 5%, after irradiation with a dosage of 99kGy. The E-modulus rises by 8%, after irradiation with a dosage of 66 kGy at the temperature 23°C and at the temperature 80°C the tensile strength rises by 7%, after irradiation with a dosage of 198kGy. A very important point is the improvement of the PA11 specimens' temperature stability, after irradiation. The tested polymer, when irradiated by a dosage of 198 kGy showed no dimensional changes - even at a temperature of 220°C. This significantly moves the application possibilities of the PA11 specimens we tested to an area with service temperatures much higher than their former melting-point. The measurements of hardness showed that irradiation also partly affects the PA11 hardness. Irradiation is also a method which reduces the creep behaviour of the studied polymer. The resistance to creep of cross-linked PA11 specimens increase with the irradiation dose.

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