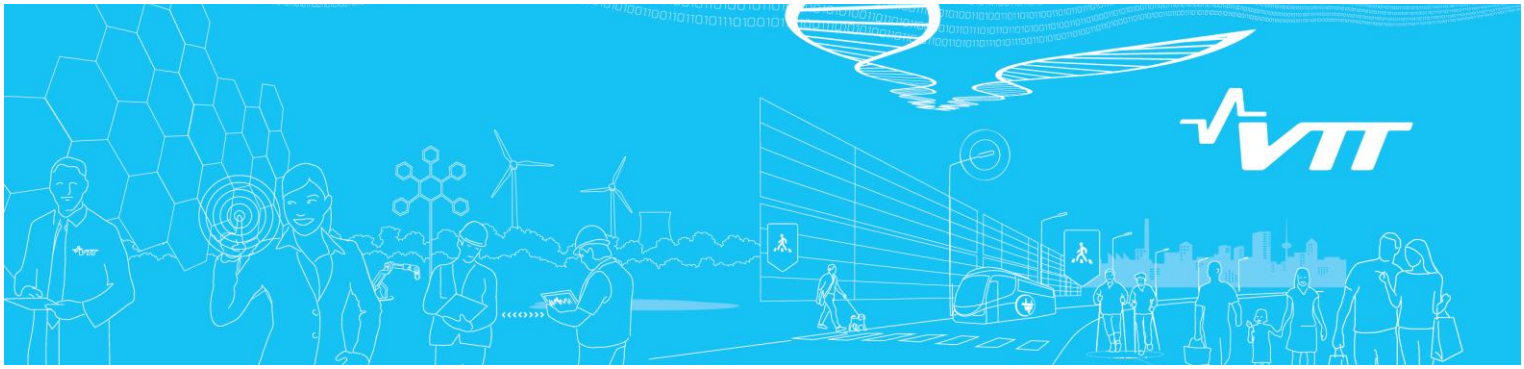

RESEARCH REPORT

VTT-R-01052-18

Dose rate effect study on EPDM and Lipalon cable jacketing materials

Authors: Konsta Sipilä, Harri Joki, Anna Jansson

Confidentiality: Public

Report's title

Dose rate effect study on EPDM and Lipalon cable jacketing materials

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Summary

Dose rate effect is a phenomenon where the damage caused to a polymer due to ionizing radiation is depending on the dose rate used during the irradiation. The degradation kinetics is such that lower dose rates cause more damage to the polymer than higher dose rates. This may pose a threat when accelerated ageing of polymers are conducted and the ageing is supposed to be equivalent to the condition that the component would be after experiencing the real service environment over many years. In order to study the existence of dose rate effect on EPDM rubber and Lipalon cable jacketing experimental data was gathered in irradiation environment and semi-empirical power law model was applied. It was noted that radiation resistivity of EPDM was rather high which impaired quality of the obtained DED (Dose to Equivalent Damage) values. In case of Lipalon it seemed that there is a dose rate effect over the dose rate range studied but in order to confirm this observation, more data on lower dose rate range would be required. Overall, it should be stated that confirming the homogenous oxidation of the irradiated samples would increase the reliability of the extraction of the DED parameters. ToF SIMS technique seemed to be sensitive in detecting ageing products but uncertainty remains how e.g. impurities of the surface affect the normalized measurement signal.

Confidentiality	Public

Espoo 27.2.2018

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Preface

This study was made as part of the project “Condition Monitoring, Thermal and Radiation Degradation of Polymers inside NPP Containments (COMRADE)” executed within the SAFIR 2018 research program. The purpose of this study was to study dose rate effect and oxidation behaviour as function of dose rate on materials that are used in Nordic nuclear power plants. Finnish State Nuclear Waste Management Fund (VYR), Swedish Radiation Safety Authority, Energiforsk Ab and VTT Technical Research Centre of Finland Ltd are acknowledged for funding this work.

Espoo 27.2.2018

Authors

Contents

1. Introduction

Polymers are known to be susceptible to dose rate effects [Gillen et al. 1981, Reynolds et al. 1995, Placek et al. 2003], where the dose rate has an effect to the amount of degradation that the polymer is experiencing. Usually the situation involves a lower dose rate causing more damage to the material than a high dose rate. This kind of scenario can be problematic when the ageing of polymeric components is simulated with accelerated ageing where high dose rates are applied, possibly yielding in too low damage levels compared to a real service environment where irradiation dose is absorbed during a period of several years.

The mechanism governing the dose rate effect is strongly related to the diffusion of oxygen which has effect to the kinetics of the chemical reactions governing the ageing of polymers. The principle of the occurrence of dose rate effect is presented schematically in Figure 1, where the dose to equivalent damage (DED) parameter is plotted against dose rate. DED parameter is defined with an end-point criterion which represents the condition of the polymer. Typical end-point criterion with cable materials is 50% absolute elongation of break but other criteria can be used as well, as long as they validly describe the condition of the polymer in its designed application. DED parameter is the absorbed dose at which the end-point criterion is reached, and as it is plotted against dose rate as illustrated in Figure 1, the dose rate effect can be visually examined.

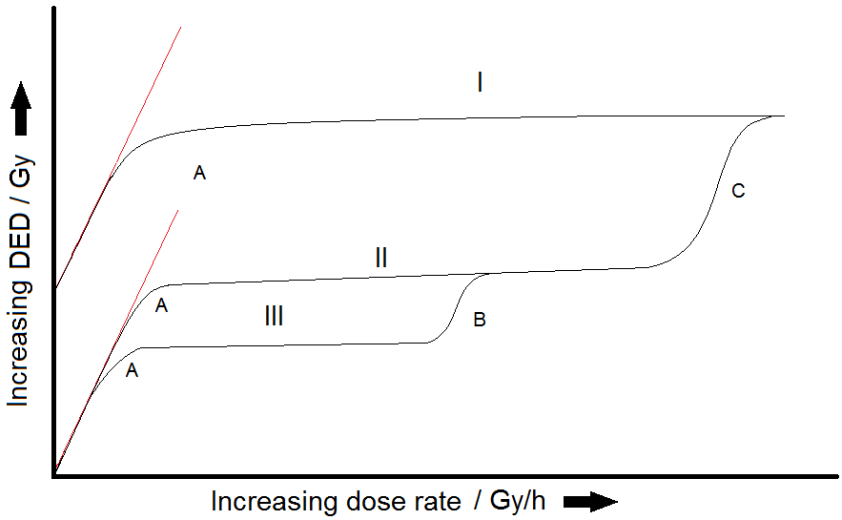

Figure 1. Schematic illustration of the dose rate effect.

Curve I in Figure 1 represents a situation where the polymer is irradiated in an inert atmosphere. The DED parameter is rather constant on very wide range of dose rates. Only at the low dose rates, a small curvature can be seen in the figure. During this curvature a small dose rate effect may take place, depending on the governing thermal and radiation degradation pathways [Gillen et al. 1993]. As the dose rate approaches zero value, the plot has slope of 1 which is the thermal ageing limit, i.e. dose rate at this stage is diminishingly small and only thermal ageing governs the ageing.

Curve II represents a situation where the irradiation is conducted in air atmosphere. In this case the effects of diffusion limited oxidation (DLO) can be seen at very high dose rates. DLO is closely related to the diffusion of oxygen and the ionizing radiation radicalizing the diffused oxygen. The interaction between an oxygen molecule and a high energy gamma quantum may result in oxygen radicals in polymer matrix which react with surrounding polymer chains ultimately yielding in chain scission or crosslinking. As the dose rate is high, the oxygen is consumed in the vicinity of the surface of the polymer and thus the degradation

of the polymer is concentrated on these surface layers. This heterogeneous oxidation can be less detrimental to the polymer than homogenous oxidation, where the oxygen has time to diffuse evenly in the whole volume of the polymer and thus degrade the polymer in wider volume.

Figure 1 has also a third curve which represents more uncommon dose rate effect that occurs at medium dose rates. This is thought to be related to the rate-limiting steps of the oxidation chemistry. Radicals having a long lifetime can be trapped inside of crystalline areas or breakdown of intermediate hydroperoxide species is sluggish enough that their effect to the degradation is not observed during the relatively short ageing treatment and following material testing period.

Studying the dose rate effect requires thus experimental data obtained in irradiation environments. The DLO effects can be studied at relative high dose rates. If the experimental data is sufficiently broad (data obtained also in thermal environments) and good quality, certain semi-empirical models may be applied and estimates on the severity of dose rate effect provided.

As DLO is closely related to the diffusion of oxygen, techniques that can detect oxidation on sample materials are convenient to use to confirm homogenous ageing of samples. Previously different techniques have been tested on oxidation profile measurements [Sipilä et al. 2017] and the Time of Flight Secondary Ion Mass Spectroscopy analysis (ToF SIMS) was considered the most promising method to measure oxidation gradients on EPDM. The measurement procedure with ToF SIMS needs to be defined in more detail, including e.g. proper sample preparation, in order to be able to produce accurate measurement results with the technique.

2. Goal

The goal was to study how the used dose rate affects to the mechanical properties of EPDM and Lipalon. In case of EPDM, it was also studied how surface oxidation is affected by dose rate.

3. Methods

3.1 Samples

The studied materials included peroxide cured EPDM rubber manufactured by James Walker and CSM cable jacket material (tradename Hypalon, cable tradename Lipalon) provided from storage of the Finnish nuclear power company TVO. The EPDM samples were stamped out of two millimetre sheet delivered by the manufacturer and Lipalon samples were prepared from the jacket of the cable delivered by TVO.

3.2 Ageing of samples

The ageing of samples was conducted by subjecting them to high dose gamma radiation. The used dose rates and absorbed total doses are shown in Table 1. The irradiation treatments having the dose rate ≥ 0,36 kGy/h were conducted at ROZA irradiation facility in ÚJV Řež, Czech Republic. The low dose rate irradiations (0,06 kGy/h) were conducted at VTT's gammacell. Both facilities use ^{60}Co as gamma radiation source. The deviation between the absorbed dose and target dose was ±10%. Same deviation applies to target and measured dose rates.

Table 1. Target doses and dose rates compared to total absorbed doses and dose rates during irradiation ageing. ToF SIMS samples marked with an asterisk.

Ageing condition #	Target dose/kGy	Target dose rate/kGy/h
1*	1000	6
2	1000	2
3*	1000	1
4	600	2
5	600	1
6	600	0,6
7	400	2
8	400	1
9	400	0,6
10*	200	2
11	200	1
12	200	0,6
13*	200	0,36
14*	200	0,06
15*	40	2

16	40	1
17	40	0,6
18*	40	0,36
19*	40	0,06

3.3 Tensile testing

Tensile testing was conducted according to ISO 37 standard. From each ageing condition five samples were tested and the tensile stress and elongation at break values were extracted from the stress-strain curves. All measurements were conducted at room temperature and strain rate of 25 mm/min was used.

3.4 Hardness measurements

Shore-A hardness was measured from base of the tensile testing samples according to ISO 7619-1 standard.

3.5 Density measurements

Density measurements were conducted by using the Archimedes' approach.

3.6 SEM analysis

Proper fracture surfaces were obtained with specimens fractured at very low temperature achieved with the application of liquid nitrogen. The fracture surfaces of the tensile specimens were analysed with scanning electron microscope (SEM).

3.7 ToF SIMS

The concentration of oxygen on the surface and in the bulk material was compared by using ToF-SIMS (Time of Flight Secondary Ion Mass Spectroscopy) which is a very surface sensitive analysis technique. The material is bombarded by a pulsed ion beam and secondary ionized molecular fragments are emitted from the surface and analyzed in the TOF (Time of Flight) detector and a mass spectrum is achieved. The spectrum can also be visualized as images as seen down to the right in Figure 2.

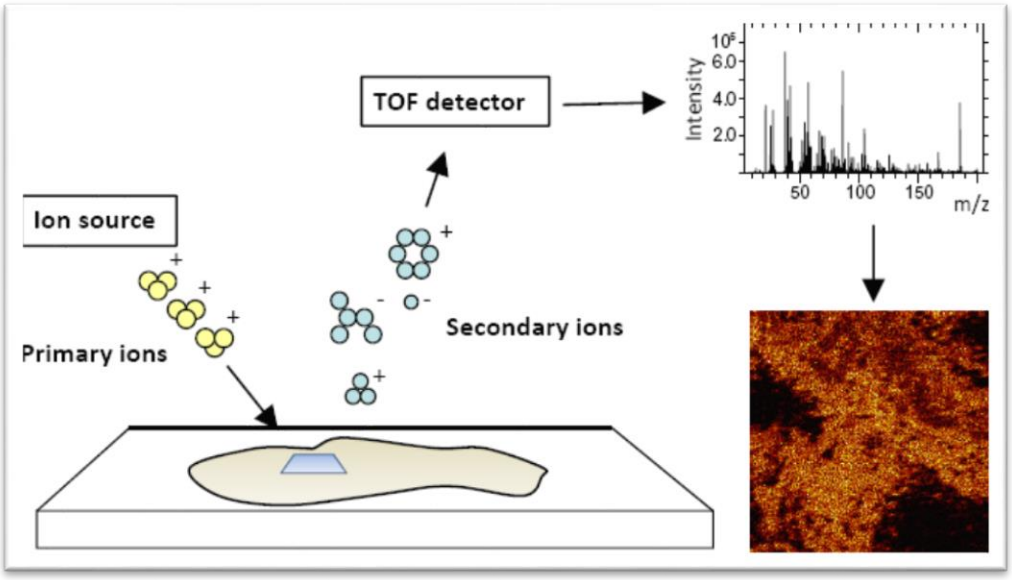

Figure 2. Illustration of the ToF-SIMS technique.

In this study the primary ions were focused on a number of positions on the material instead of imaging mode (used in 2016 studies, reported in [Sipilä et al. 2017]), where the ion beam was moved in a raster pattern over a cross section.

The material was exposed to gamma radiation and samples were extracted and analyzed after different doses of radiation according to Table 1 (samples marked with an asterisk). In addition to the irradiated samples, one reference sample was analyzed.

Since the test matrix was rather large (eight samples) and ToF-SIMS is a time consuming analyzing technique it was decided that the analyses were performed on the sample surface and in the center of a cross section, approximately 2 mm from the surface, instead of scanning the cross section and using the imaging technique. The samples were washed by ethanol and acetone before analyzes to remove dirt and other contaminants from the surfaces.

In the first set of runs rubber samples turned out to be difficult to prepare for the analysis and it was difficult to achieve a flat surface and hence the analyses were difficult to interpret [Sipilä et al. 2017]. Extra caution was taken during the sample preparation in order to avoid uneven and contaminated surfaces. Ethanol was used to lubricate the material and the cutting tool.

4. Results and discussion

4.1 Mechanical properties of EPDM

4.1.1 Tensile testing

The tensile testing results for EPDM are shown in Table 2. Elongation of break and tensile strength values at different dose rates and as a function of the absorbed dose are shown in Figure 3 and Figure 4, respectively.

Table 2. EPDM tensile testing results.

Ageing condition #	Absorbed dose/kGy	Dose rate/kGy/h	Elongation at break/%	Tensile strength/MPa
0	0	0	182	13,0
1	1000	6	90	10,1
2	1000	2	80	9,7
3	1000	1	77	10,8
4	600	2	115	10,8
5	600	1	134	10,6
6	600	0,6	108	10,5
7	400	2	145	11,3
8	400	1	142	10,6
9	400	0,6	126	9,4
10	200	2	162	11,4
11	200	1	158	11,4
12	200	0,6	160	11,7
13	200	0,36	152	12,0
14	200	0,06	158	12,7
15	40	2	213	13,7
16	40	1	191	11,8
17	40	0,6	182	11,4
18	40	0,36	206	12,5
19	40	0,06	197	12,3

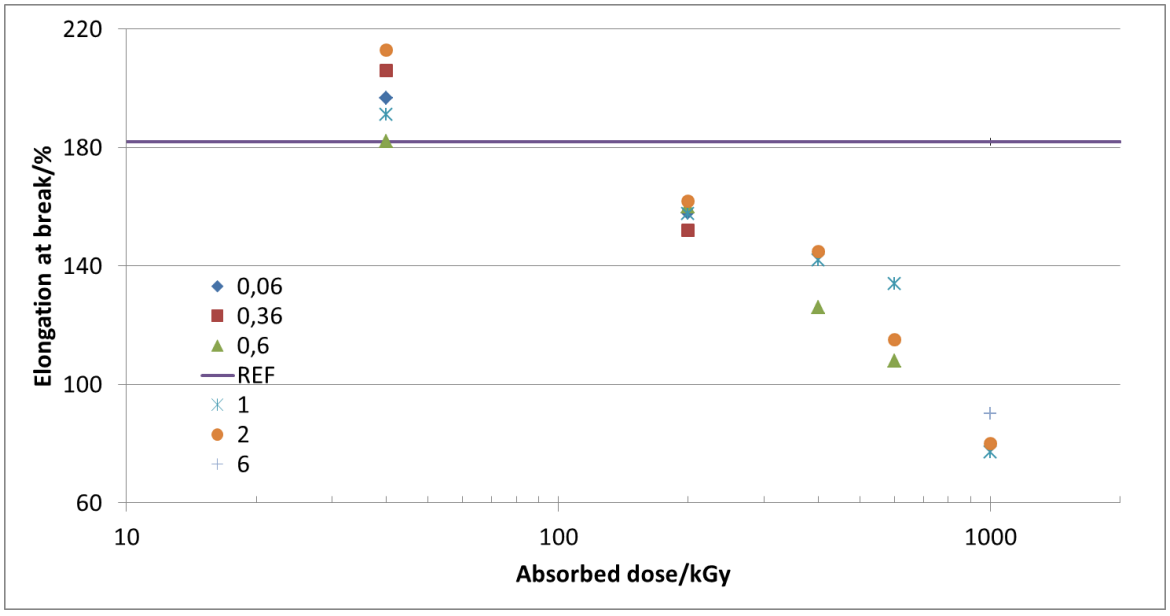

Figure 3. Elongation of break of EPDM at different dose rates as function of absorbed dose.

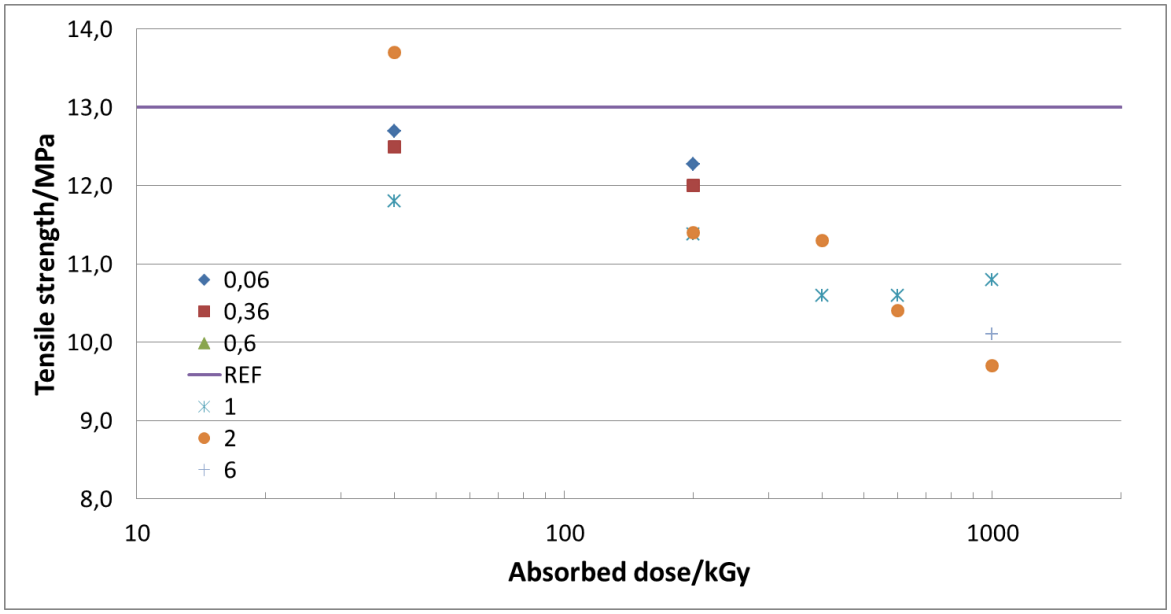

Figure 4. Tensile strength of EPDM at different dose rates as function of absorbed dose.

Both elongation of break and tensile strength decrease as the absorbed dose increases. No clear indications between different dose rates can be extracted from the data presented in this form. In order to predict existence of the dose rate effect, extraction of DED values (the dose at which the end-point criterion is met) was conducted according to the power law model [IEC TS-61244-2]. In this case, the typical 50% absolute elongation of break end-point value is not used since it was not simply reached during ageing. Also by defining multiple end-point criteria, the sensitivity of the power law model can be examined. The end-point criterion is defined as the relation of measured and initial elongation of break, e/e_0. DED values can be extracted when the end-point criteria at different dose rates are plotted versus the absorbed dose, as shown in Figure 5. While extracting the data, the linear function was fitted to the experimental data and it was used to extrapolate the low end-point criteria. All extracted DED values are gathered in Table 3. For dose rate of 0,06 kGy/h no values are presented since the incompletion of the data.

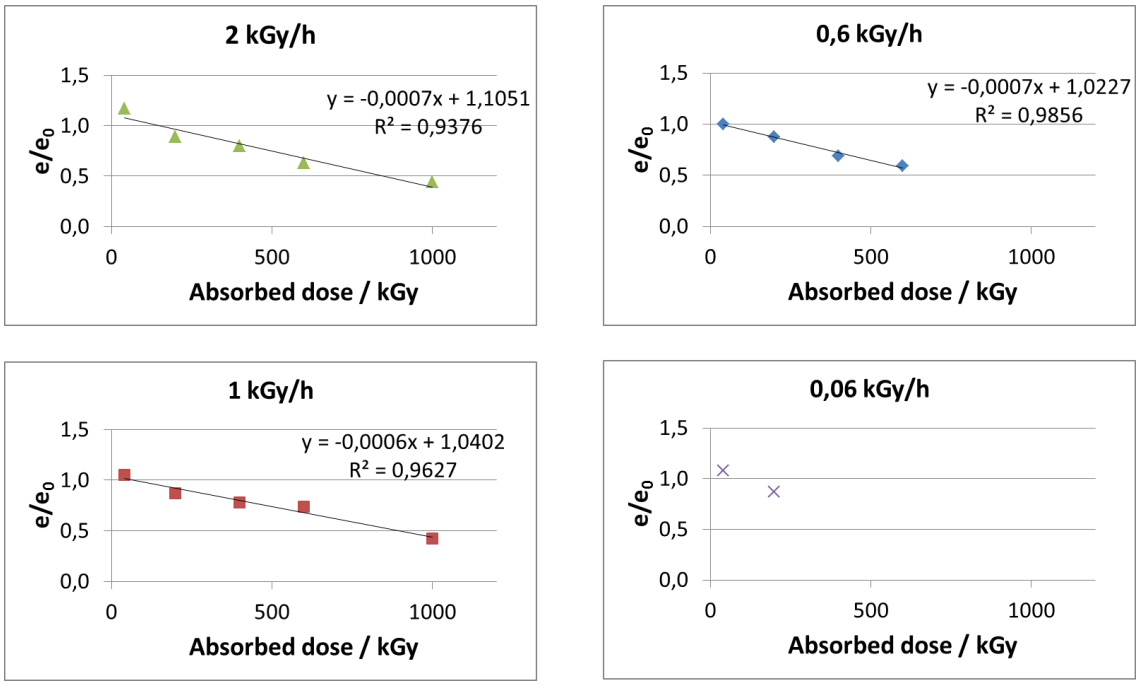

Figure 5. Extraction of end-point criteria from experimental data.

Table 3. The absorbed doses in kGys at each end-point (i.e. DED values) as function of dose rate.

	Dose rate / kGy/h		
e/e_0	**0,6**	**1**	**2**
0,9	175	234	293
0,8	318	400	436
0,7	461	567	579
0,65	532	650	650
0,6	604	734	722
0,5	747	900	864
0,4	890	1067	1007
0,3	1032	1234	1150
0,25	1104	1317	1222

The dose at which end-point criteria is met can be plotted versus the dose rate. When this is done on a logarithmic scale, DED values can be predicted from a straight line that follows equation:

$$DED = KD^n \quad (1)$$

Where *D* is the dose rate, *K* and n specific material parameters, where *n* is typically between 0 and 0,4. In Figure 6 the part of the data from Table 3 is illustrated graphically and fitting is done according to equation 1. Thus the DED values can be predicted to lower dose rates.

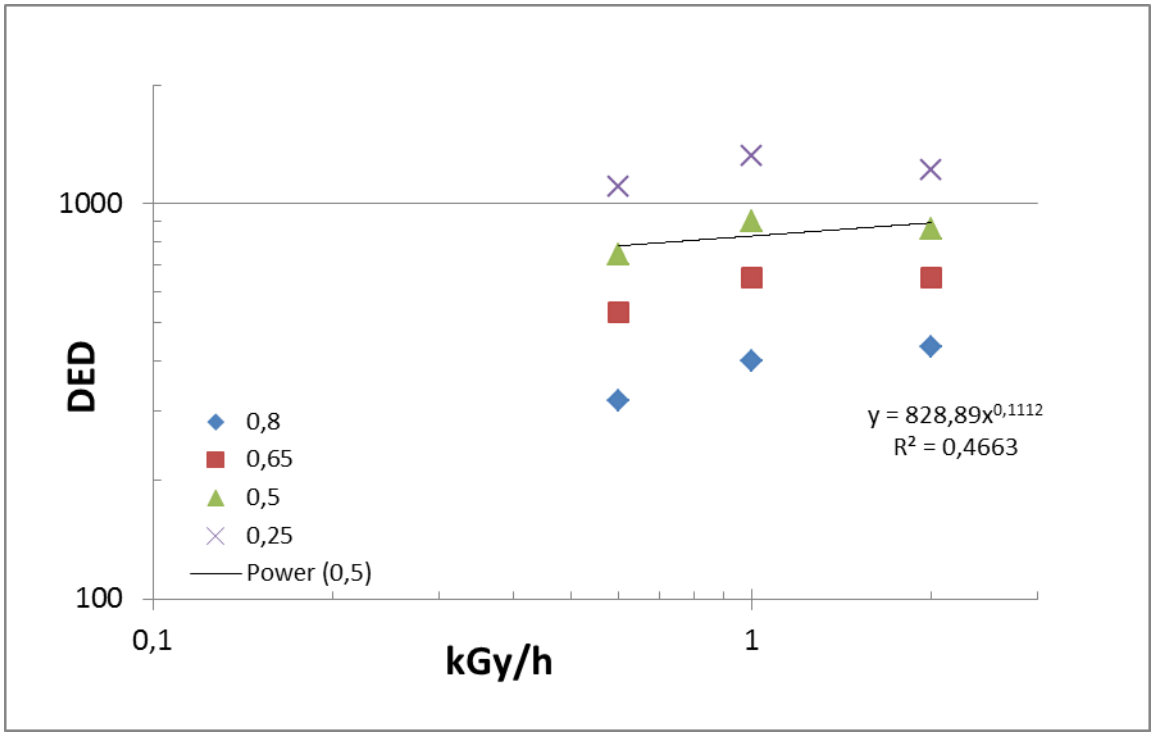

Figure 6. Extrapolation of end-point dose to lower dose rates for EPDM.

The closer examination of Figure 6 reveals that the obtained fits for the data sets are not good (e.g. R-squared value for 0,5-data set shown in Figure 6). This is thought to conclude

from heterogeneous oxidation of the samples at high dose rates which yields different ageing mechanism between the data sets obtained at different dose rates. Also in the case when using “predicted” end-point (i.e. 0,25) some discrepancy may be included in the extrapolation.

4.1.2 Hardness measurements

Hardness measurement results are shown in Table 4. Hardness was not measured for all samples since from the previous experiments [Sipilä & Joki, 2017] it was noticed that significant amount of absorbed gamma radiation is required in order to introduce an observable increase in the measured Shore A hardness. The results indicate a slight increase in hardness as the absorbed dose becomes 1000 kGy. There is a three-unit difference in hardness on these 1000 kGy samples as the dose rate is decreased from 6 kGy/h to 1-2 kGy/h. Otherwise, the variations in hardness are two units or less when constant doses are plotted as a function of dose rate, indicating that the hardness increases only a little when the absorbed dose is less than 1000 kGy.

Table 4. Hardness measurement results for EPDM.

EPDM LR9444 total dose [kGy]	**6 kGy/h**	**2 kGy/h**	**1 kGy/h**	**0,6 kGy/h**	**0,36 kGy/h**	**0,06 kGy/h**	**0 kGy/h**
1000	76	79	79				
600		73		75			
400		73		73			
200		72			72	74	
40		70				71	
0							73

4.1.3 Density measurements

The density measurement results are shown in Table 5. The general trend seems to be that the density slightly increases as the absorbed dose is increased to 1000 kGy even though density seems to be quite constant on absorbed doses from 40 to 600 kGy but still a bit higher than the measured reference value. The maximum increase in density was 1,5 % (in case when total dose was 1000 kGy and dose rate 1 kGy/h) as the maximum decrease was 1,1 % (in case when total dose was 1000 kGy and dose rate 6 kGy/h). There are only two samples where density has decreased from the initial value, one being the sample with 1000 kGy absorbed dose and the very high applied dose rate of 6 kGy/h. This is also a bit different from the other two samples irradiated with 1000 kGy dose and might indicate a variation in ageing mechanism. However, this should be confirmed by defining the oxidation products on the samples. Other explanations to the changing densities could be scission and crosslinking of polymer chains as well as decomposition and/or evaporation of different additives.

Table 5. Density measurement results for EPDM (g/cm^3).

EPDM LR9444 total dose [kGy]	6 kGy/h	2 kGy/h	1 kGy/h	0,6 kGy/h	0,36 kGy/h	0,06 kGy/h	0 kGy/h
1000	1,086	1,112	1,114				
600		1,096		1,104			
400		1,101		1,101			
200		1,104			1,106	1,107	
40		1,103				1,103	
0							1,098

4.1.4 SEM analysis

SEM images were taken from a reference sample and a sample that had been irradiated to the dose of 1000 kGy (illustrated in Figure 7 and Figure 8, respectively). The overall difference between the surfaces seems to be that the irradiated sample has rougher surface than the reference sample.

Figure 7. Fracture surface of EPDM reference sample.

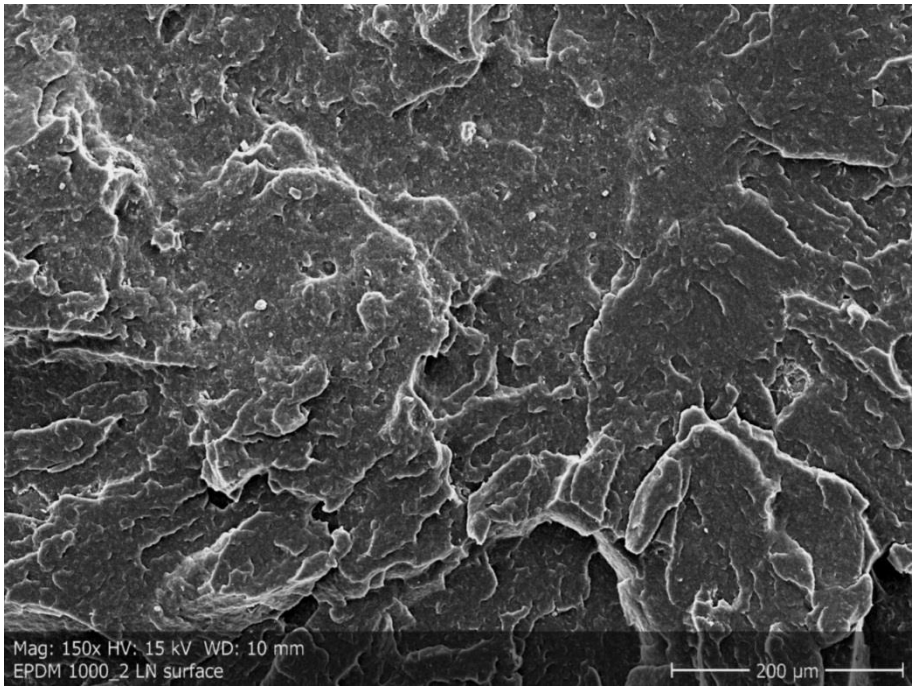

Figure 8. Fracture surface of EPDM sample after irradiation of 1 MGy absorbed dose.

4.2 Mechanical properties of Lipalon

4.2.1 Tensile testing results

Tensile testing results are shown in Table 6. Elongation of break and tensile strength results at different dose rates are plotted as function of absorbed dose in Figure 9 and Figure 10, respectively.

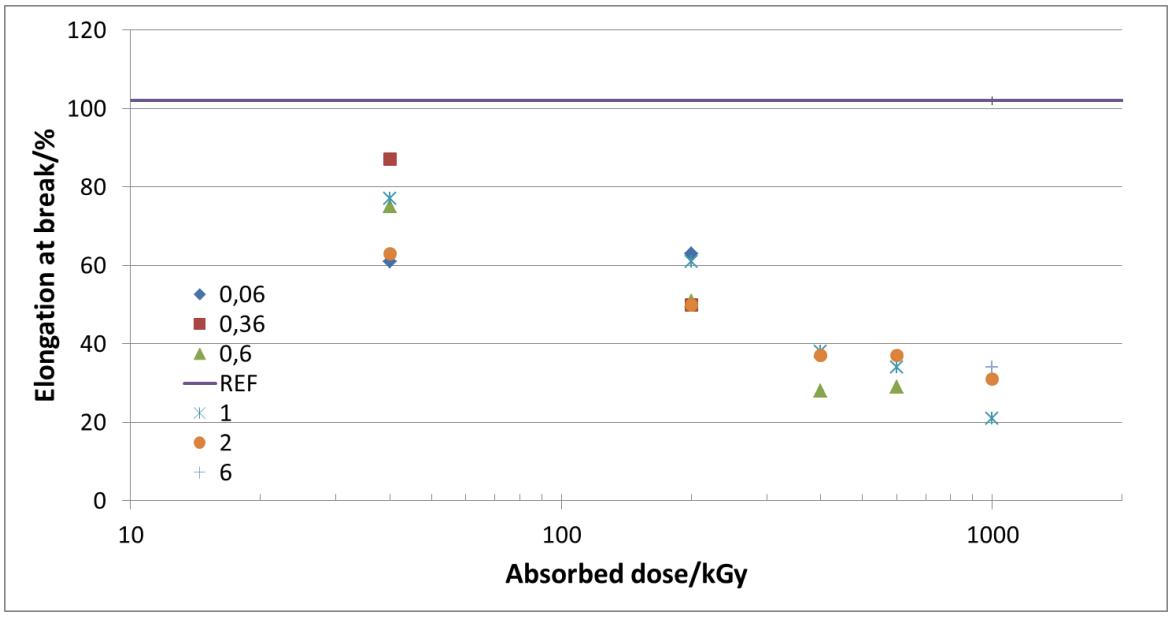

Figure 9. Elongation of break of Lipalon at different dose rates as function of absorbed dose.

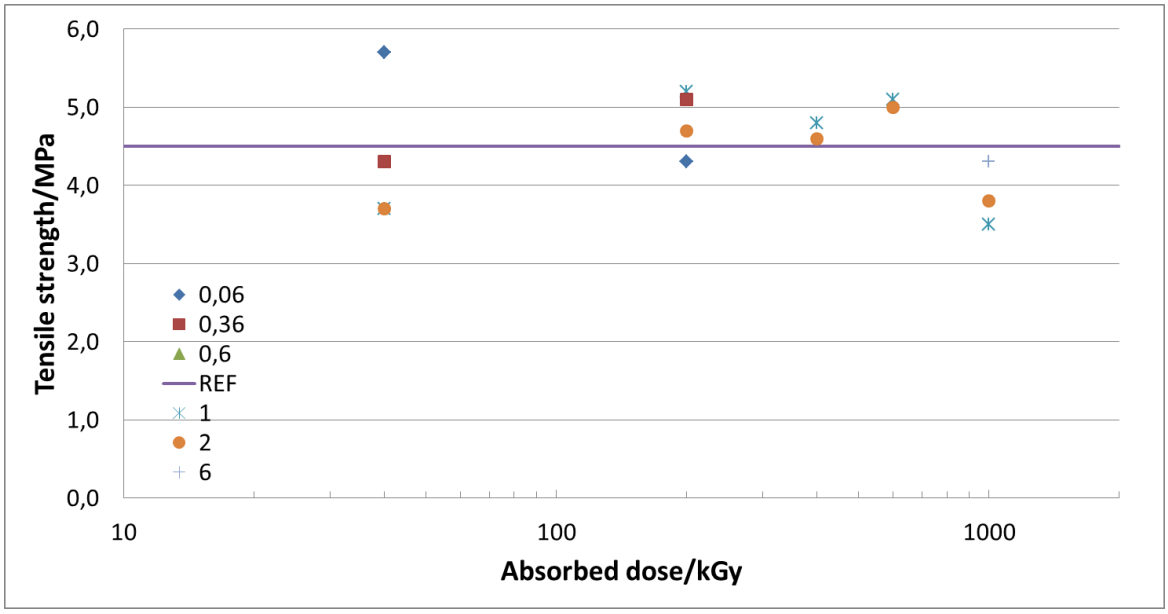

Figure 10. Tensile strength of Lipalon at different dose rates as function of absorbed dose.

Table 6. Tensile testing results for Lipalon.

Ageing condition #	Absorbed dose/kGy	Dose rate/kGy/h	Elongation at break/%	Tensile strength/MPa
0	0	0	102	4,5
1	1000	6	34	4,3
2	1000	2	31	3,8
3	1000	1	21	3,5
4	600	2	37	5,0
5	600	1	34	5,1
6	600	0,6	29	5,2
7	400	2	37	4,6
8	400	1	38	4,8
9	400	0,6	28	4,2
10	200	2	50	4,7
11	200	1	61	5,2
12	200	0,6	51	5,3
13	200	0,36	50	5,1
14	200	0,06	63	5,7
15	40	2	63	3,7
16	40	1	77	3,7
17	40	0,6	75	3,8
18	40	0,36	87	4,3
19	40	0,06	61	4,3

The elongation of break values decrease until absorbed dose of 400 kGy is reached and after that, the general trend seems to be that no significant changes in elongation of break can be observed, with the exception of the samples irradiated with dose rate of 1 kGy/h. There are no significant changes in tensile strength values as function of absorbed dose. Decrease in elongation of break combined to constant tensile strength values as function of increasing absorbed dose indicate embrittlement of the material. DED values related to different end-point criteria were extracted from Figure 11 and these DED values are shown in Table 7.

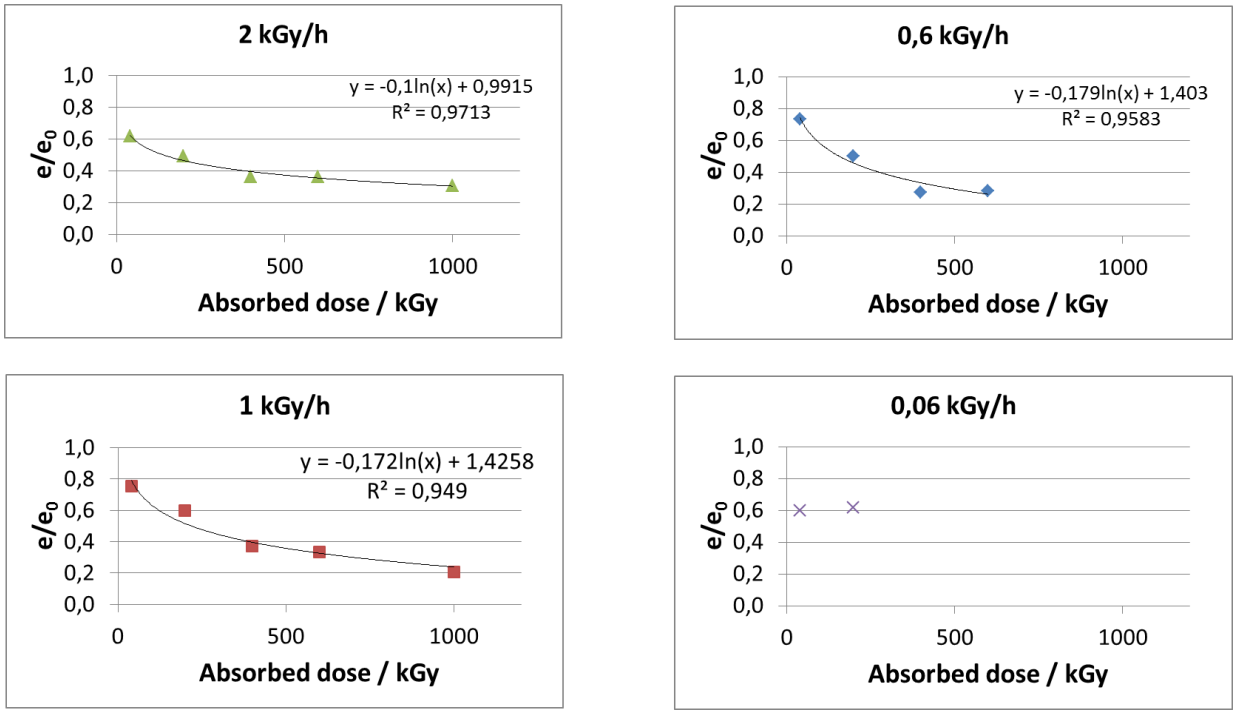

Figure 11. Extraction of end-point criteria from experimental data.

Table 7. The absorbed doses in kGys at each end-point (i.e. DED values) as a function of dose rate.

	Dose rate / kGy/h		
e/e_0	**0,6**	**1**	**2**
0,9	17	21	2
0,8	29	38	7
0,75	38	51	11
0,7	51	68	18
0,6	89	122	50
0,5	155	218	136
0,4	271	389	371
0,3	474	696	1007
0,25	627	931	1661

The extrapolated DED values are shown in Figure 12. It seems that according to equation 1 an excellent fit is obtained when low end-point criterion is used (i.e. 0,25 and 0,3). The obtained fit gets worse as the end-point value is increased. This may be due to the large scatter of the tensile testing of the high end-point results. The overall trend seems to indicate

that there is a dose rate effect as the dose rate decreases. However, it seems that more data obtained with lower dose rates would be required to make more conclusive deductions since the used dose rates in this study are relatively high.

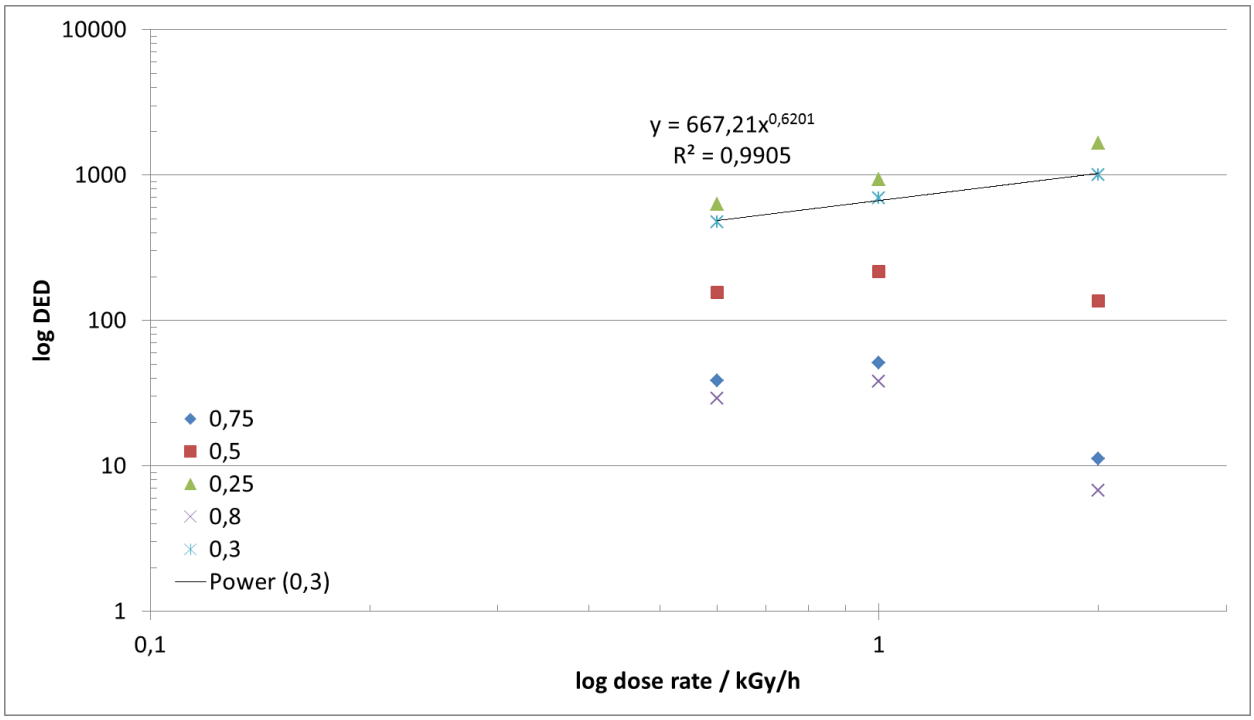

Figure 12. Extrapolation of end-point dose to lower dose rates for Lipalon.

4.2.2 Hardness measurements

The hardness results for Lipalon samples are shown in Table 8. The hardness of Lipalon samples is steadily increased as the absorbed dose increases. However, the effect of dose rate is not so straightforward. When the absorbed dose is 600 kGy, the increase of hardness is five hardness units as the dose rate is decreased from 2 kGy/h to 0,6 kGy/h. Thus it seems that there could be some kind of effect originating from dose rate but it should be confirmed whether the trend continues at lower dose rates. In addition, at lower absorbed dose of 200 kGy where such data exists, no similar conclusions could be stated. Increasing hardness along with the decreasing elongation of break indicate the embrittlement of the material.

Table 8. Hardness measurement results for Lipalon samples as function of dose rate at different absorbed doses.

Total dose / Dose rate	**6 kGy/h**	**2 kGy/h**	**1 kGy/h**	**0,6 kGy/h**	**0,36 kGy/h**	**0,06 kGy/h**	**0 kGy/h**
1000	76	75	79				
600		76		81			
400		75		73			
200		72			72	70	
40		68				70	
0							67

4.2.3 Density measurements

The density measurement results for Lipalon are shown in Table 9. The lowest measured value was 2,3% lower than reference (400 kGy absorbed dose and 2 kGy/h dose rate) and highest value was 1,1% higher than the reference (40 kGy absorbed dose and 2 kGy/h dose rate). Generally, the measured values are lower than the reference value, with the exception of samples that absorbed 40 kGy dose. The sample irradiated with 200 kGy dose with 0,06 kGy/h dose rate is also higher but in such extent that the deviation may be included in the error margin. Decrease in the density may be interpreted to conclude from increased crosslinking. This yields in increasing amounts of side chains which decreases the density since structure that has less side chains can be packed in smaller volume.

Table 9. Density measurement results [kg/dm^3] for Lipalon samples as function of dose rate at different absorbed doses.

Total dose / Dose rate	**6 kGy/h**	**2 kGy/h**	**1 kGy/h**	**0,6 kGy/h**	**0,36 kGy/h**	**0,06 kGy/h**	**0 kGy/h**
1000	1,494	1,501	1,500				
600		1,486		1,500			
400		1,476		1,491			
200		1,505			1,498	1,512	
40		1,526				1,520	
0							1,510

4.2.4 SEM analysis

SEM images were taken from a reference sample and a sample that had been irradiated to the dose of 1000 kGy (illustrated in Figure 13 and Figure 14, respectively). Similar effect was observed in the Lipalon samples as in the case of EPDM, since the irradiated specimen showed more rough fracture surface than the reference sample.

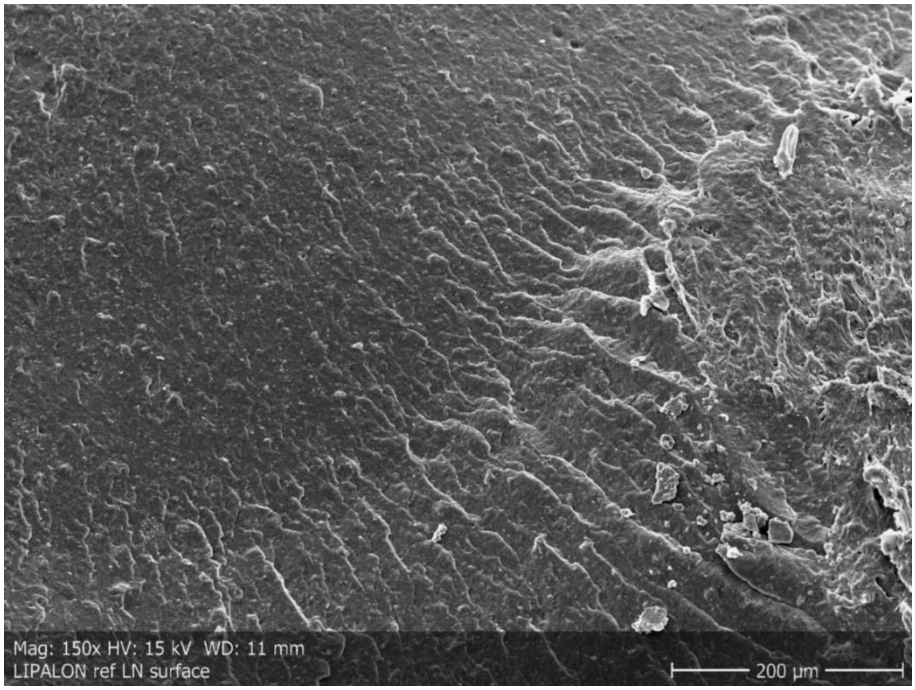

Figure 13. Fracture surface of Lipalon reference sample.

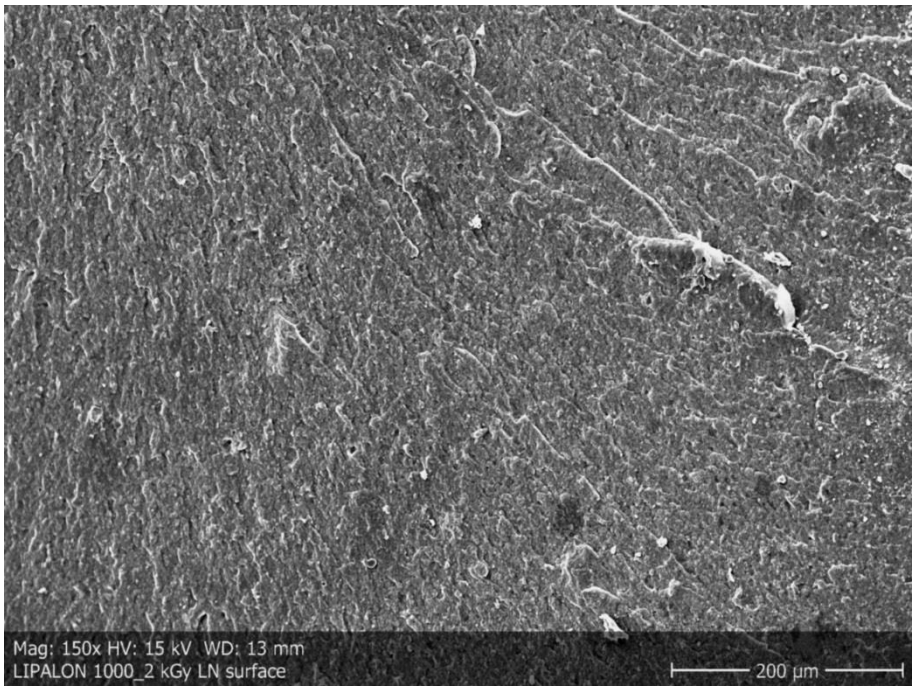

Figure 14. Fracture surface of Lipalon sample after irradiation of 1 MGy absorbed dose.

4.3 Oxidation profiles of EPDM measured with ToF SIMS

The results are shown in Figure 15 below. The signals are normalized to the total amount of two fragments (CH and C_2H) which exist both on the surface and in the bulk material.

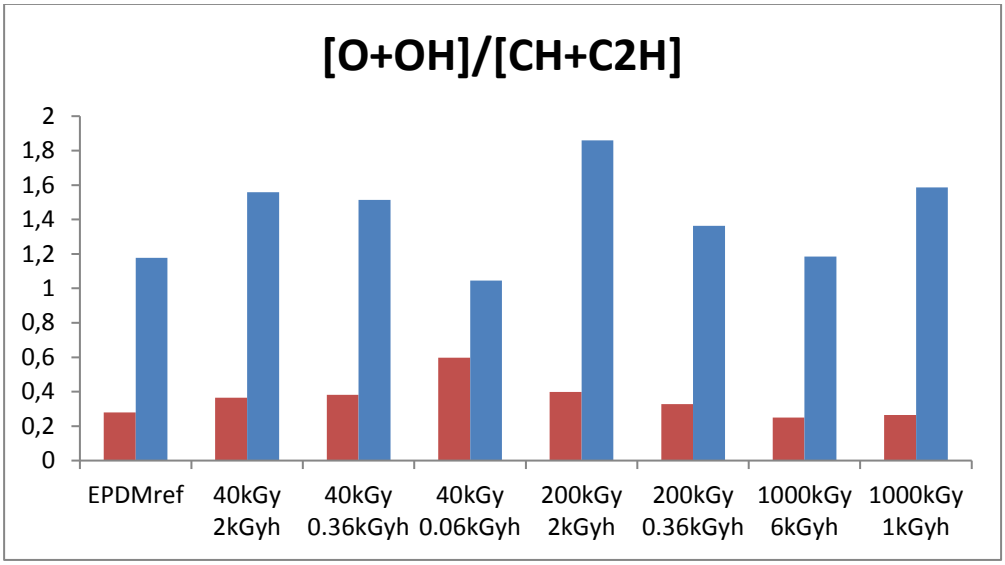

Figure 15. The red bars show total oxygen content in the bulk material and the blue bars the surface material. The surface seems to contain higher concentration of oxygen containing reaction groups.

However, there is a risk of over-estimating the ion concentration on the surface due to handling and contamination on the surface and insufficient cleaning of the samples, the so called yield effect.

As for the other results (tensile testing, hardness etc.) no significant correlation of oxidation and dose rate effect can be seen. Generally the oxygen content is clearly lower in bulk, expect in the case of 40 kGy/0,06 kGy/h where the difference between bulk and surface is less than a half. This kind of behavior would be expected as the dose rate is decreased and oxygen has time to diffuse further into the bulk before being radicalized by ionizing radiation. When dose rates 2, 0,36 and 0,06 kGy/h are compared, 0,06 kGy/h shows the lowest oxygen content on the surface and this might indicate the formation of crosslinks instead of formation of oxygen containing degradation products at higher doses of gamma radiation. In order to verify this, samples irradiated with 0,06 kGy/h and higher absorbed doses should be analyzed.

5. Conclusions

The ageing of EPDM and Lipalon samples were studied as function of absorbed dose and dose rate. The surface and bulk oxidation of EPDM was studied by using ToF SIMS. EPDM showed good radiation resistance which yielded in uncertain predictions of dose rate effect when the power law model was applied. In case of Lipalon the dose rate had and an effect to the DED values but more experimental data from low dose rate irradiations would be required in order to confirm this observation. Overall, it should be stated that the used dose rates during the irradiations were relatively high and homogeneity of oxidation could not be confirmed which would ease the examination of the data quality. ToF SIMS seemed to be very sensitive technique in detecting oxidation products in the samples but identifying artefacts from the normalized signals still causes uncertainty to the measurement results.

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