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Procedia Structural Integrity 2 (2016) 2405–2414

Structural Integrity Procedia

www.elsevier.com/locate/procedia

21st European Conference on Fracture, ECF21, 20-24 June 2016, Catania, Italy

# The study of temperature and radiation induced degradation of cable polymers: A comparison between the mechanical properties of industrial and neat EPDM

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**Abstract**

The mechanical properties of industrial and neat Ethylene Propylene Diene Monomer (EPDM) polymers, aged under $\gamma$-irradiation at different temperatures, are studied. The focus is given to the dose rate effects in polymer insulation materials, so the ageing is performed in the wide range of dose rates, doses and temperatures. Industrial EPDM samples are extracted from the cables in use in Belgian Nuclear Power Plants (NPP), and the neat EPDM samples are produced in the laboratory. The mechanical tests of non-aged and aged polymers are performed, and the methodology of estimating the polymer life time is discussed. The ultimate tensile stress and elongation at break are found to be strongly affected by both irradiation condition and temperature. The ultimate tensile stress clearly exhibits the dose rate effect observed through the shift of the crossover between cross-linking to chain scission process as a function of the dose. This crossover shifts to high dose for large dose rates, while the opposite is observed by increasing the temperature. Dose rate effect is less evident in the elongation at break data, probably because both cross-linking and chain scission affect the elongation at break in the same way, by decreasing it. In comparison to industrial EPDM aged under the same conditions, the cross-linking to chain scission crossover appears at lower dose in neat polymer and the elongation at break decreases faster by increasing the dose. In addition, the elongation at break experimental results can be modeled by changing single parameter, namely pre-exponential factor of the irradiation rate constant. This confirms that both aging processes, cross-linking and chain scission affect the elongation at break in a similar way, by deteriorating network structure responsible for polymer elastic properties. Irradiation rate constant is found to follow the square root dependence for industrial EPDM, while the linear dependence is observed for the neat EPDM. This indicates the existence of different degradation processes in these two polymers.



*Keywords:* polymer degradation; mechanical properties; radiation and thermal ageing; EPDM

## 1. Intorudction

The lifetime extension of existing nuclear power plants (NPP) relies on validation of the safe long term performance. For that purpose it is of outmost importance to demonstrate the structural and functional integrity of the NPP constituents for extended time usage. Unlike some other components, the monitoring program for cables has not been established, so NPP industry expressed the need for accelerated cable ageing experiments, additional testing and analysis of cable properties.

Cable ageing studies are often focusing on the cable insulation, since the loss of insulation might lead to the loss of cable functionalities. Cable insulation is typically made of polymers like polyethylene (PE), polyvinyl chloride (PVC), ethylene-propylene elastomers (EPR, EPDM), or cross-linked polyethylene (XLPE). Some cables which are used in


10.1016/j.prostr.2016.06.301

Belgian NPPs are made of ethylene-propylene-diene monomer (EPDM terpolymer) mixed with additives and slightly cross-linked, in order to improve their mechanical and physical properties. Thue (2011.); Zuidema et, al (2011.); Mead at al. (2002.)

Degradation mechanisms caused by $\gamma$-irradiation and elevated temperatures of the EPDM polymers are extensively studied. Two main ageing mechanisms responsible for mechanical and physicochemical material changes are the cross-linking and chain scission Rivaton et al. (2004., 2005,.). During the ageing, these two processes occur simultaneously, and their mutual contributions to the polymer degradation depend on many factors, e.g. polymer composition, ageing conditions and aging environment. The chain scission is stabilized in oxidizing environment since alkyl radicals created during the ageing process strongly react with oxygen. The chain scission is thus particularly important at elevated temperatures which facilitates the oxygen diffusion and increases reactivity of free radicals Rivaton et al. (2005.); Planes et al. (2010.). Moreover, accelerated ageing of polymers in oxidizing environment may introduce the dose rate effect which originates from diffusion-limited heterogeneous polymer oxidation. Dose rate variation will also create different oxidation rates, so the polymer might oxidize less for the same total dose Gueguen et al. (1994.); Pinel et al. (1999., 1994.). This effect could promote the cross linking. In order to have valid extrapolation from accelerated ageing conditions to NPP relevant conditions, the effects related to the temperature and dose rate have to be clarified.

Consequences of thermal and radiation ageing can be monitored through the change of polymer mechanical properties. The ultimate tensile stress is observed to increase due to the cross-linking, and to decrease in the case of the chain scission, which makes this parameter very sensitive to the dose rate effect. The dose rate effect is typically manifested by the opposite behaviors of the ultimate tensile stress and elongation at break as a function of irradiation dose Clough and Gillen (1985.). In particular, at high dose rates elongation is observed to decrease drastically while the ultimate stress remains constant by increasing dose. On the contrary, at low dose rates the elongation at break retains the high values while the ultimate tensile strength drops rapidly. In the paper of T. Seguchi *et al.* Seguchi et al. (1981.) it was shown for the cross-linked polyethylene that the elongation at break and ultimate tensile strength at a given dose are all dependent on the dose rate in a way that change is enhanced at low dose rates. In cross-linked EPR, this effect was observed to be suppressed for the elongation at break.

An additional complexity related to an interplay of cross-linking and chain scission arises from the use of different additives, causing the great variation of the EPDM responses to the ageing, in particular in industrial polymers. Because of that, there is still no full understanding of EPDM polymer degradation processes. In this study, the analysis of aged industrial and neat EPDM polymer is presented. The focus is given to the mechanical properties of these polymers aged in the wide dose and temperature ranges.

## 2. Materials and Methods

The industrial samples used in this study are extracted from the ERR cable polymer installed in a Belgian NPP. Main polymer matrix component is EPDM, named Nordel 2722 with a ethylidene norbornene (ENB) as a diene, and it is present 80 pbw (parts by weight). EVA polymer is also present with 20 pbw. As flame retardant filler aluminium hydroxide (ATH) is used and amount of it is very high, 101 pbw. Crystallinity of samples, measured with differential scanning calorimetry (DSC), is found to be 0.7 weight percent. The samples were punched out from a cable with a 2 mm thick dumbell shape. Dumbbell dimensions are according to the dimensions for the small dumbbell test piece from the IEC International standard 60811-1-1 IEC International standard 60811-1-11 (2001.), so the total length was 50mm, length of the narrow part 17 mm, and the width of the narrow part is 4 mm.

Polymer named NORDEL IP 3722 for the production od neat samples was kindly provided by DOW chemicals. This polymer is designed to match flow properties of Nordel 2722 Snyder (2000.), which is not in production anymore. Nordel IP 3722 is a semi-crystalline polymer, with low diene (ENB) 0.5 weight percent. Crystallinity reported by the producer is 15 wt %. This material is specially designed for medium and low voltage wire and cable applications.

Neat polymer is delivered in pellet form. In order to get the adequate samples, 2 mm thick sheets were made and the dumbbell samples were punched out of them using the same knife system as for industrial EPDM. Neat EPDM polymer sheets were produced by pellets melting and pressing. Obtained polymer sheet was considered to be good if boundaries within melted pellets were not noticeable to the naked eye. Appropriate melting temperature and pressing time are important with respect to the sheet quality and they were established to be $160\ ^0C$ and 3 minutes.

In the frame-like mould shape about 55g of pellets was spread over. This system was then placed in melting machine and it was melted for 5minutes while slightly pressed. To get compact sheet pressing is done right after melting under pressure of 8-10 bar. Dumbbell samples were punched out with sample dimensions matching the dimensions of industrial EPDM samples.

Ageing of sample was done at the Belgian research center SCK·CEN by performing simultaneous thermal and irradiation ageing. The irradiation was performed in the RITA or BRIGITTE irradiation facility described in details previously Fernandez et al. (2002.). The RITA facility comprises four 20 cm high cylindrical $Co^{60}$ sources located at $\sim$ 6.5 m below the water surface. The experimental samples are placed in the water-tight irradiation container. To perform exposure to gamma-radiation this container is lowered into the underwater position in the center of the square formed by the $Co^{60}$ sources. The size of the square can be changed to adjust the dose-rate. The BRIGITTE facility is similar to RITA with a difference that it comprises up to 10 $Co^{60}$ sources and the dose-rate adjustment is done by changing the number of sources.

In both facilities the gamma-sources have a length shorter than the container height. As a result the dose-rate inside the container has a vertical gradient with a flat dose-rate area around the mid-plane. A desirable dose-rate can be obtained by changing the vertical position in the irradiation container. The actual dose-rate at the sample location was mapped before the irradiation using Harwell Red Perpex dosimeters. The standard accuracy of the dosimetry is 5.5%. The non-uniformity of the dose-rate distribution over the samples was less than 5%. When under irradiation, the samples were located in a dedicated cylindrical furnace placed inside the irradiation container to allow for accurate temperature control throughout the experiment. This furnace is a thick wall Al tube with closed ends and a heating wire placed on the outside surface and covered with a thermal insulation. A high thermal conductivity of Al allows for a reasonably quick thermal response with a time of $\sim$ 40 min to achieve stabilization at a desired temperature within $\pm 0.1^0C$. The temperature under irradiation is maintained using a PID controller. The volume of this furnace is $\sim$ 20 l. There was no air supply to the furnace: the irradiation was performed in stagnant air.

Typically 10 samples with total mass of about 10 g are used for one irradiation condition. Since the EPDM oxygen consumption is about $6.24 \times 10^{-5}$ mol/g at 10kGy Arakawa et al. (1987.), the total oxygen consumption by the samples during irradiation for doses up to 1000kGy is less than 1% of the total volume. Accordingly, irradiation is not performed in depleted oxygen conditions. On the other hand, the critical sample thickness, relevant for oxygen limited diffusion on the basis of IEC report 61244-1 IEC Technical Report 61244-1 (2014.), is found to be Lc $\sim$ 1.65 mm for our irradiation conditions (the air pressure of about 1.3 Bar). This value is slightly below the typical sample thickness of about 2 mm used in this study, so on the basis of this evaluation one could also expect limited contribution from heterogeneous oxidation.

The aging matrix is given in Table 1. The aging temperature is in a range of $25^0C$ to $85^0C$, and the dose rate from 106 Gy/h to 1390 Gy/h.

Table 1. Ageing matrix of the industrial EPDM samples.

| Dose rate ($Gy/h$) | Absorbed dose ($kGy$) | Ageing temperature ($^0C$) |
|---|---|---|
| 250 | 100 200 300 400 500 600 | 25 |
| 720 | 200 300 400 500 600 700 800 900 1000 1100 | 25 |
| 1390 | 200 300 400 600 800 1000 | 40 |
| 2760 | 317 466 793 1189 1586 2379 | 40 |
| 106 | 20 40 60 100 200 250 | 55 |
| 455 | 80 160 240 400 800 1000 | 55 |
| 106 | 20 40 60 100 200 250 | 70 |
| 455 | 80 160 240 400 800 1000 | 70 |
| 1390 | 200 300 400 600 800 1000 | 70 |
| 2760 | 320 470 800 1200 1600 2400 | 70 |
| 106 | 20 40 60 100 200 250 | 85 |
| 455 | 80 160 240 400 800 1000 | 85 |

Table 2. Ageing matrix of the neat EPDM samples.

| Dose rate ($Gy/h$) | Absorbed dose ($kGy$) | Ageing temperature ($^0C$) |
|---|---|---|
| 450 | 52 76 129 194 258 388 | 40 |
| 1400 | 160 235 400 600 800 1200 | 40 |
| 2780 | 320 470 801 1201 1601 2402 | 40 |
| 450 | 50 75 125 200 250 400 | 70 |
| 1400 | 160 235 400 600 800 1200 | 70 |
| 2780 | 320 470 801 1201 1601 2402 | 70 |

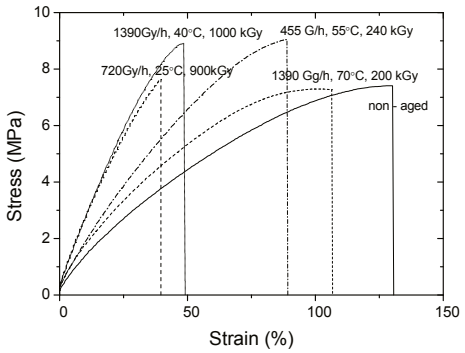


Fig. 1. The engineering stress versus engineering strain of industrial EPDM polymer.

The tensile test is performed on a Lloyd LR10K machine, at a speed of 10 mm/min at room temperature ($22^0C$). The samples were clamped at 8 mm from each side and the crosshead displacement and force as a function of time were measured. The displacement was additionally recorded optically. Two samples were tested per one ageing condition.

## 3. Results and discussions

Differences in mechanical properties between industrial EPDM and neat EPDM (in further text referred to as NORDEL) were initially compared on engineering stress versus engineering strain curves. Examples of typical stress versus strain curves of the non-aged and aged industrial EPDM polymer are shown in Figure 1, while the stress - strain curves of non-aged and aged NORDEL polymer are presented in Figure 2. Clear difference is observed in the tensile behaviour and ultimate tensile values. Industrial EPDM has significantly lower elongation at break value compared with the NORDEL. Middle strain value is 125.5 ±10 % for NORDEL and 975 ±20 %. This was expected knowing that industrial polymer contain the additives that inhibit chain mobility and extension properties.

The tensile curve of EPDM shows that the stress increases monotonously, in a non-linear way, up to the failure point. This behaviour is typical of elastomers. The fact that the fracture occurs already in the region of monotonous behavior, indicates the existence of brittle (less ductile than expected) behavior, as it should be expected for industrial EPDM type of polymers Seidel (2008.). Yield point is observed in the tensile curve of non-aged NORDEL as a consequence of its semi-crystalline structure. For semi-crystalline polymers, the necking mechanism involves orientation and destruction of semi-crystalline morphologies, resulting in the existence of the yield pointAnderson (2005.). In EPDM polymers, polyethylene unites are responsible for the appearance of semi-crystalline morphologies. Polyethylene in the neat polymer (NORDEL) has more freedom to arrange into crystalline zones, but in industrial EPDM, the presence of second polymer (EVA), fillers (ATH) and other additives inhibit polyethylene chain parts to arrange into more ordered structure. Still, one can see that for high doses the yielding point disappears in NORDEL tensile curves. Most probably this effect occurs since the crystalline structure is destroyed, but this remains to be confirmed by physicochemical experiments.

The appearance of necking during the mechanical testing of NORDEL samples is illustrated in Figure 3. While an industrial EPDM after rupture completely recovers it's initial shape, the NORDEL samples irradiated to low dose ex-

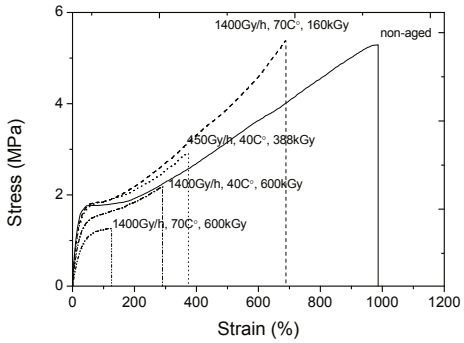


Fig. 2. The engineering stress versus engineering strain of neat EPDM polymer ( Nordel).

Fig. 3. Necking behaviour observed in neat polymer aged to low dose (white samples). The total recovery in industrial EPDM (black samples).

hibit macroscopic plastic deformation/necking. On the contrary, at high irradiation dose both NORDEL and industrial EPDM brake in elastic regaion of the stress strain curve.

The effects of dose rate and temperature are analyzed by observing the change of ultimate tensile versus total dose. In Figure 4, the ultimate tensile stress data for industrial EPDM are presented. By increasing the dose, the ultimate tensile stress first increases and then decreases, exhibiting a local maximum. According to the literature, this maximum corresponds to the transition (cross-over) between the regime where cross-linking dominates, and the regime where chain scission dominates the degradation mechanisms Rivaton et al. (2005); Gueguen et al. (1994.); Cellete aet al. (2004.); Seguchi et al. (1983.). The ultimate tensile stress of the industrial EPDM samples, aged at different temperatures at the medium dose rates is shown in Fig.4a). By increasing the ageing temperature the cross linking region is gradually suppressed, and the local maximum is shifted to a lower dose.

Dose rate effect, or the shift of the cross-over as a function of the dose is also analyzed at low Figure 4b) and high ageing temperatures Figure 4c). For the samples aged at low temperatures, e.g. in the range of 25-40 $^0C$, an increase of the dose rate leads to the slight gradual shift of the cross-over to higher dose. With respect of the samples aged at high temperatures, e.g. in the range of 75-85 $^0C$, the dose rate effect is observed to be much stronger. The shift of cross-over is not more evidenced at low dose rate of about 106 kGy/h. Generally, one ca say that cross-linking region (increase of the ultimate stress) is promoted at high dose rates.

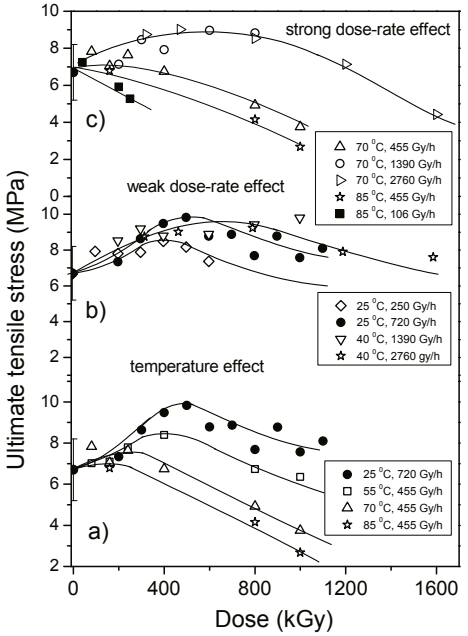


Fig. 4. The change of ultimate tensile stress as a function of dose for industrial EPDM aged at a) different temperatures for middle range of dose rates b) different dose rates in the low temperature range c) different dose rates in the high temperature range. The full line is a guide for an eye.

Products of polymer macromolecules bonds rupture, caused as a consequence of polymer - $\gamma$ energy interaction, are known as macro- radicals. Upon creation, macro-radicals may undergo mutual interaction and create new bonds. This process is known as cross-linking. However, macro-radicals are very sensitive to oxygen presence, they can react fast with oxygen by creating peroxy radicals and hydroperoxides. These products can further react with the polymer chain causing chain scission (rupture of original bonds), or react with one another and terminate oxidation process. Which process will be dominant depends on macro-radicals concentration, mobility, but also on oxygen concentration in the vicinity of the radicals. Generally, the high production of radicals (high dose rate) and low oxidation will promote the cross-linking, while the high temperature (increased oxygen diffusion) and long exposures in oxidized conditions (usual conditions when achieving the high dose at low dose rates) will promote the chain scission. The crossover appears when number of oxidized bonds overcome number of cross-links. The fact that the chain scission overcomes the cross-linking effect above certain dose has been confirmed earlier Rivaton et al. (2005,.); Cellete aet al. (2004.).

In Figure 5 the stress results of NORDEL and industrial EPDM are compared to each other. The comparison is shown for the ageing temperatures of $40^0C$ (Fig.5a) and $70^0C$ (Fig.5b). Slightly lower stress value for the non-aged NORDEL in a comparison with non-aged industrial EPDM is observed. This is expected, since NORDEL has no fillers and it was not cross-linked by peroxides during the sample preparation process. The crossover between the cross-linking and chain scission is clearly seen for NORDEL samples aged at $40^0C$, but it occurs at lower dose in comparison with the industrial EPDM. For NORDEL aged at $70^0C$, the crossover is further shifted to lower doses, following the same trend as in industrial EPDM. In addition the decrease of the ultimate stress as a function of dose for the samples aged at $70^0C$ is faster in comparison with NORDEL aged at $40^0C$. Dose rate effect is also observed in NORDEL material . Cross-linking to chain scission crossover at low doses has been confirmed earlier for non-industrial polymers. The crossover in neat EPDM films that are irradiation aged is measured through an increase in gel fraction as a function of the irradiation dose and found to be of about 100 kGy Rivaton et al. (2005). The relaxation measurements by Celette et al. Cellete aet al. (2004.) on 100 $\mu$m thick samples indicate the transition to be at about 200 kGy. Our mechanical measurements agree well with these results. Since in industrial EPDM crossover appears at much higher dose at about 600 kGy, this indicates that industrial polymer is more chemically stable.

In the previous studies reported the existence of two distinct regimes for the ageing at 40 $^0C$, and for dose rates between 10 - 2500 Gy/h up to 100 kGy: an ultimate stress decay for low dose rates (10 and 100 Gy/h) and ultimate

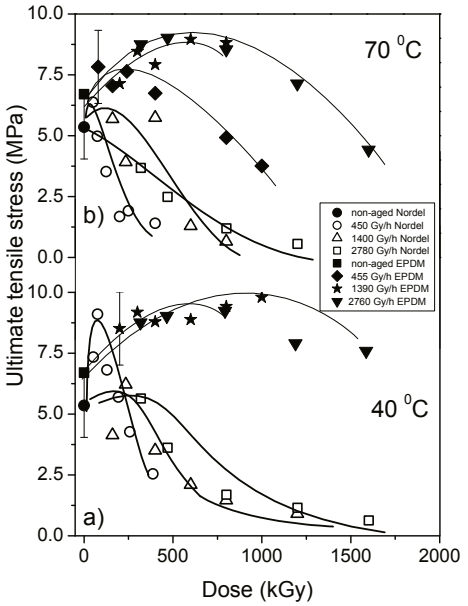


Fig. 5. The comparison of the ultimate tensile stress values for industrial EPDM and NORDEL aged at a) $40^0C$ b) $70^0C$. The full line is a guide for an eye.

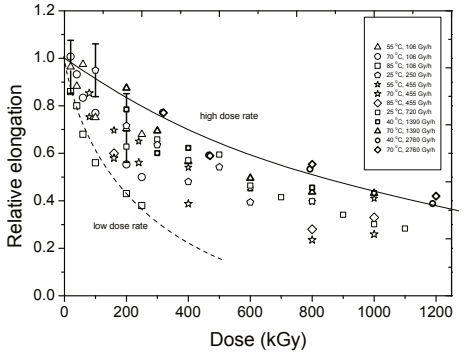


Fig. 6. The change of elongation at break in industrial EPDM. The lines are guide for an eye.

stress increase for dose rates higher than 500 Gy/h Gueguen et al. (1994.). Similar observation is also reported by Pinel and Boutaud Pinel et al. (1999., 1994.). Our results rather show that the crossover gradually changes, while effect of interplay of dose rate and ageing temperature is confirmed. While increase of the temperature shifts the transition to lower dose, an increase of dose rate shifts the transition to higher dose. The existence of a crossover is essentially observed in entire dose range considered in this study.

Change of elongation at break as a function of dose for the industrial EPDM is presented on the Figure 6. Dose rate effect is found to be less pronounced, as it has been observed earlier, Seguchi et al. (1981.). Still, for extreme ageing conditions clear difference in elongation at break decrease is evidenced. So, for the samples aged at high ageing temperatures and low dose rates elongation at break decreases much faster in comparison with samples aged for very high dose rates.

The comparison of the elongation at break of the industrial EPDM with those of NORDEL (Fig7.) as a function of dose shows that elongation at break decreases faster for neat EPDM. Once again this effect can be assigned to the absence of fillers and stabilisers which make NORDEL less chemically stable. Dose rate effect is observed to be stronger at high temperatures. The fact that the elongation at break is less sensitive to the dose rate effect could be

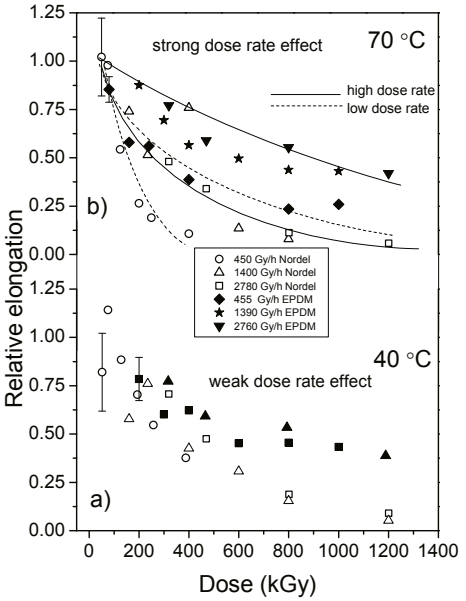


Fig. 7. The comparison of the elongation at break values for industrial EPDM and NORDEL aged at a) $40^0C$ b) $70^0C$. The lines are guide for an eye.

the consequence of the fact that cross-linking and chain scission both contribute to the decrease of the elongation at break.

The relative elongation as a function of the ageing time are often analyzed on the basis of model developed by Menlow and Dunkin Pinel et al. (1994.):

$$e/eo = \{(1 + (\beta - 1))Kt)\}^{(1/(1-\beta))} \tag{1}$$

where $K = K_{th}e^{(-E_{ath}/(k_bT))} + K_{irr}e^{(-E_{airr}/(k_bT))}$ is the reaction rate constant including Arrhenius (activation) type thermal and irradiative contributions. The beta is the overall order of the degradation process. The $E_{ath}$ and $E_{irr}$ are thermal and irradiative activation energies, respectively.

The comparison between the experimental results and calculations based on Eq. 1 is presented for the industrial EPDM (Figure 8a), and NORDEL (Figure 8b).

Very good agreement between calculated and measured elongation at break data was found for both materials. Essentially, all experimental results could be reproduced with Eq.1 by varying the single parameter, pre-exponential factor of the irradiation rate constant, $K_{irr}$. All other parameters from Equation 1 were kept constant in the calculation. They are collected in Table 3. Both thermal and radiative activation energies, as well as the thermal reaction rate constants were found to be in a very good agreement with previous results Pinel et al. (1994.).

Table 3. The parameteres used in the calculation according to the model described by Equation 1.

| Parameter | Value industrial EPDM | Value neat EPDM |
|---|---|---|
| $E_{ath}$ | 1.07 eV | 1.07 eV |
| $K_{th}$ | $50\times10^9$ $h^{-1}$ | $50\times10^9$ $h^{-1}$ |
| $E_{irr}$ | 0.065 eV | 0.065 eV |
| $\beta$ | 3.5 | 2 |

The$\beta$ parameter is obtained to be 3.5 and 2 for industrial EPDM and NORDEL, respectively. In both materials the elongation at break is governed by $K_{irr}$ parameter in a similar way. By increasing the dose rate the $K_{irr}$ parameter increases monotonously and no irradiation temperature effect is observed for the industrial EPDM. Behavior of $K_{irr}$ of industrial polymers is analyzed on the basis of $K_{irr} = CI^{\alpha}$, where I is dose rate, alpha is dose rate exponent and C is a constant. It is found that the $K_{irr}$ follows a square root dependence ($\alpha = 0.5$) as a function of the dose rate. This result agrees well with the dose rate dependance of the oxygen consumption Seguchi et al. (1983.). Accordingly, the

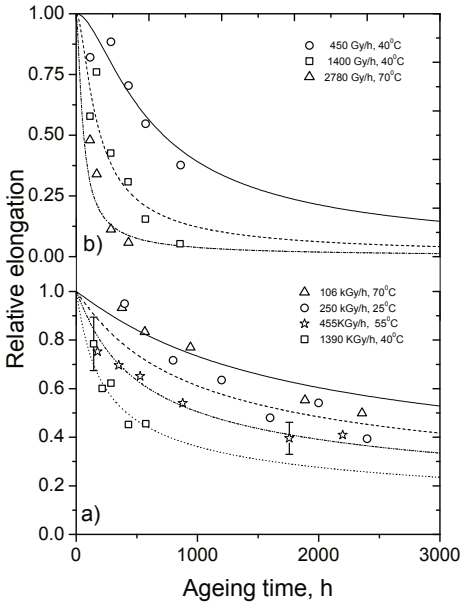


Fig. 8. The relative elongation as a function of the ageing time for a) industrial EPDM and b) Nordel.

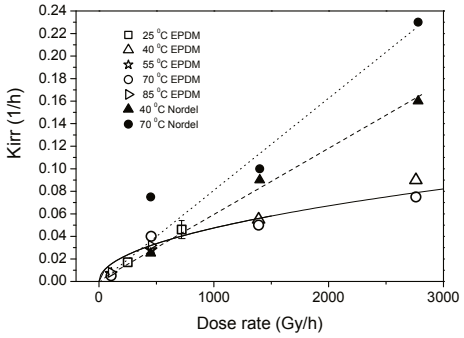


Fig. 9. Radiative reaction rate constant as a function of the dose rate.

elongation at break of industrial EPDM is mainly governed by the oxidation process. On the contrary, the $K_{irr}$ value of the neat polymer as a function of dose seems to be dependent on the temperature, especially for the lowest and the highest dose rates. The value of $K_{irr}$ of NORDEL samples is higher in comparison with industrial EPDM, and the differences increases by increasing the dose rate. NORDEL samples aged at samples aged at $40^0C$ and $70^0C$ exhibit linear dependence of $K_{irr}$ as a function of the dose in contrast to square rood dependence observed for industrial EPDM. This indicates that that there is a difference in the degradation mechanisms of industrial and neat polymer. While irradiation and temperature assisted oxidation effect play the major role in EPDM, the chain scission caused by irradiation seems to be main degradation mechanism in neat polymer (NORDEL). The dose rate effect is less observed in elongation at break data in comparison with the ultimate tensile stress, possibly because both cross-linking and chain scission have the same effect on the elongation at break via destruction of network structure which is responsible for elongation properties. Because of that, the total elongation can be used as the relevant parameter for the cable life time prediction.

## 4. Conclusion

In this study the analysis of aged industrial EPDM polymer used in Belgian NPP cables, as wellas the neat EPDM (NORDEL) polymer is performed on the basis of the mechanical tests. The materials are aged in a wide range of temperatures and dose rates. Elongation at break and the ultimate tensile stress are found to be strongly affected by the irradiation dose. In contrast to the ultimate stress which showed clear dose rate effect, this effect is less evident in the elongation at break data. NORDEL polymer is found to be less chemically stable for the same ageing conditions. With respect of the modeling, all elongation at break experimental results can be reproduced by the model based on the change of single parameter, namely pre-exponential factor of the irradiation rate constant.

## Acknowledgements

The authors would like to thank Prof. J. Devaux, Prof. T. Pardoen and T. de Schoutheete for valuable discussions. The authors acknowledge the support from the GDF-SUEZ-ENGIE under contract No: 045

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