**EFFECTS OF RADIATION TYPES AND DOSE RATES
ON SELECTED CABLE-INSULATING MATERIALS**F. Hanisch¹⁾, P. Maier²⁾, S. Okada³⁾ and H. Schönbacher⁴⁾**ABSTRACT**

A series of radiation tests have been carried out on halogen-free cable-insulating and cable-sheathing materials comprising commercial LDPE, EPR, EVA, and SIR compounds. Samples were irradiated at five different radiation sources, e.g. a nuclear reactor, fuel elements, a ⁶⁰Co source, and in the stray radiation field of high-energy proton and electron accelerators at CERN and DESY. The integrated doses were within 50 to 5000 kGy and the dose rates within 10 mGy/s to 70 Gy/s. Tensile tests and gel-fraction measurements were carried out. The results confirm that LDPEs are very sensitive to long-term ageing effects, and that important errors exceeding an order of magnitude can be made when assessing radiation damage by accelerated tests. On the other hand, well-stabilized LDPEs and the cross-linked rubber compounds do not show large dose-rate effects for the values given above. Furthermore, the interpretation of the elongation-at-break data and their relation to gel-fraction measurements show that radiation damage is related to the total absorbed dose irrespective of the different radiation types used in this experiment.

(Submitted to Radiation Physics and Chemistry)

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1. INTRODUCTION

In order to maintain the reliability and safety of electrical insulating materials which are used in areas of high radiation level, a series of radiation tests [1] are being carried out by the European Organization for Nuclear Research, CERN. Within the programme of radiation testing of cable-insulating materials [2-4], 14 different cable-insulating and cable-sheathing materials were selected in order to study the degradation effects of radiation types and dose rates. They can be divided into two groups: one is for investigation of the dependence of long-term irradiation effects on stabilizer type and concentration on extruded low-density polyethylenes (LDPEs). The second group comprises chemically cross-linked rubbers: ethylene vinyl acetate (EVA), ethylene-propylene rubber (EPR), and silicone rubber (SIR). Some of these materials contain fillers and other additives; in two cases, polymer combinations were used as polymer matrix.

All materials were available in dumb-bell-shaped tensile samples, and were irradiated at five radiation sources at different doses and dose rates. In addition to the tensile tests, gel-fraction measurements have been carried out, and the change of this chemical property is compared with the elongation at break. Finally, the dose-rate dependence of the end-point doses and the aspects of extrapolation to lower dose rates are presented.

2. EXPERIMENTS

The materials used in this investigation are listed in Table 1. For easy identification, each has been assigned a number. The materials are commercially available (522, 532), or are compounded of raw materials which are generally used in the cable industry. In the case of flame-retardant materials, the fillers are of the alumina-trihydrate type; no antimonite/halogen system was used. None of the materials contain any significant amounts of corrosive additives, so they pass the relevant tests for halogen-free cable materials. The compounds can be used for high-quality signal and power cables.

For each material a package of five tensile test samples was irradiated in nineteen positions at the five different radiation sources, as indicated in Table 2 together with doses and dose rates:

- reactor ASTRA: series (a), (b), (c), (d);
- ^{60}Co : series (e), (f);
- spent-fuel element: series (g), (h), (i);
- stray radiation from the electron-positron storage ring PETRA (DESY, Deutsches Elektronen Synchrotron, Hamburg): series (j), (k), (l), (m), (n);
- stray radiation near targets at the CERN Super Proton Synchrotron (SPS): series (o), (p), (q), (r), (s).

All doses cited in this report are ^{60}Co equivalent doses in $(\text{CH}_2)_n$ materials, e.g. polyethylene. They have been derived either by dosimetry specially adapted to the radiation source (reactor, ^{60}Co source, fuel-element facility) or by our routine radiophotoluminescence (RPL) dosimeters installed in each sample package—which is covered by a 0.3 cm thickness of the material in question—together with special dosimeters (LiF crystals, H_2 pressure) to correct for radiation quality at the accelerator positions.

Tensile test samples were made according to DIN Standard 53504 size S3. For the gel-fraction measurement, the tensile test sample was cut into pieces. These were packed into a pocket-shaped container made of stainless-steel net. The mesh size was #100 for the first group of materials (LDPEs 522 to 528) and #115 for the second group (rubber compounds 529 to 535). The solvent was Xylene for the first group, and Toluene for the second one.

3. RESULTS AND DISCUSSIONS

3.1 Results of tensile tests

The mechanical test results were obtained according to ISO R 527 and R 37. Care was taken to carry out the tests under similar conditions for comparable materials, especially for the LDPE series.

For most materials, the tensile strength test results vary with dose by less than a factor of 2. They are usually lower at the higher doses, with the exception of materials 534 (EVA elastomer) and 535 (EPR/EAR elastomer), or remain constant within measurement error. Similarly, the variations in hardness are small for the thermoplasts (less than 8 degrees change in Shore hardness); but the variations are appreciable for the cross-linked materials 531–535 — up to twice the initial value, and even a factor of 5 for silicone rubber.

The elongation-at-break measurements were, however, the most sensitive of all, displaying reductions of a factor of 50 for the highest doses. Within measurement error, no significant increase in elongation was detected. Because of the large radiation effects, only the results for elongation at break are presented in the figures for comparison of the influence of the different irradiation sources and dose rates for each material. This presentation is also in line with IEC Standard 544, Part 4, which recommends the elongation at break as the critical property for the assessment of radiation degradation of flexible insulating materials [5, 6].

A detailed comparison between the different materials will be given below. The correlation between the degradation of the elongation and the gel fraction clearly shows a dependence on the base polymer, the additives, and the chemical cross-linking. The results are discussed in subsections 3.2.1 to 3.2.3, which deal with low-density polyethylenes, polymer mixtures containing the same amino-type stabilizer, and chemically cross-linked rubber compounds, respectively.

3.2 Elongation-at-break results and their relation to the gel fraction

3.2.1 Low density polyethylenes (Materials 522 and 524 to 528)

For the irradiations in the reactor ASTRA at dose rates between 8 and 70 Gy/s, the dose dependence can be considered identical for all LDPE materials. A representative example for four LDPEs (522 and 525 to 527) is shown in Fig. 1a. The variations for differently stabilized materials are of the order of 10%; it is only at the dose of 1 MGy that they reach 35% of the mean value, which corresponds to about 100% elongation in absolute value. The results indicate that, under these conditions of high dose rates, the type of damage is different from oxidative degradation, which prevails at lower dose rates and against which the stabilizers provide protection, as they do against thermally initiated degradation. The same applies for the ^{60}Co irradiations, which were all carried out at the same dose rate of 0.64 Gy/s; the results are also shown in Fig. 1a.

After irradiation at the spent-fuel-elements facility at a dose rate of 60 mGy/s, the expected strong dependence on the amount of stabilizer has been observed at all doses (Fig. 1b). This confirms the estimate of 30 to 60 mGy/s for the upper limit [7, 8] of dose rates, which is required if these samples are to be fully penetrated by atmospheric oxygen, since the influence of stabilizers is still great. The difference in degradation due to this influence amounts to a factor of 5 at 0.2 MGy, of 3 at 0.5 MGy, and of 2 at 1 MGy.

For the irradiations near the vacuum chamber of PETRA, the dose rates vary in the range between 8 and 60 mGy/s, with some differences for the various materials within each series. At the low dose rates the gradients of oxygen concentration within our samples are expected to be small, and the influence of stabilizers is clearly seen (Fig. 1c). A dependence of degradation similar to that obtained at the spent-fuel elements is found, with more than a factor of 5 difference due to stabilization at 0.2 MGy and about 20 mGy/s. At the lowest dose rates of 4 to 8 mGy/s and a dose of about 0.05 MGy, the results are more or less comparable, for both doses and dose rates, with those

of the series irradiated at CERN. At the higher dose rates of the CERN irradiations, which cover the range between 0.05 and 1.15 Gy/s, the stabilizer influence is lower and qualitatively the same as at the ^{60}Co source.

An intercomparison of the results obtained with the five different radiation sources is shown in Fig. 1d for one selected material (LDPE 525) for which the type of stabilizer and its amount (phenolic, 0.08%) are usual for thermally stabilized LDPEs. The same results are obtained for other phenolic-type stabilizers in the same amount (e.g. material 524). A further addition of ozone protection wax gives slightly better results at low dose rates (e.g. material 526 in comparison with 528).

The results of gel-fraction measurements of all LDPEs treated in this section show a very similar tendency; the elongation, the gel fraction, and the swelling ratio have therefore been plotted in a three-dimensional presentation in Fig. 2. Data points which have typical doses (0.5 MGy and 1 MGy) and dose rates were selected. The data points (a) and (b) correspond to the reactor irradiation (high dose rate, ≈ 60 Gy/s); (e) and (f) to the ^{60}Co source (intermediate dose rate, ≈ 0.6 Gy/s); and (h) and (i) to the spent-fuel elements (low dose rate, ≈ 0.06 Gy/s). The total integrated dose for (a), (e), and (h) is 0.5 MGy, and for (b), (f), and (i), 1 MGy. The elongation clearly shows a dose-rate dependence which can be explained from the viewpoint of oxygen diffusion. This means that cross-linking is more dominant at higher dose rates, leading to high gel-fraction values. The supply of oxygen molecules diffused into the inside of the material is too low to oxidize polymer radicals formed by irradiation. Therefore cross-linking reactions between the polymer radicals predominate. At lower dose rates, on the other hand, oxygen penetrates to a considerable depth, which leads to oxidation scission reactions.

An example of the correlation between the elongation and the gel fraction is given in Fig. 3 for LDPE 524. This shows the same equivalent dose lines (solid line, 0.5 MGy and 1 MGy) and equivalent dose-rate lines (broken line, 60 Gy/s, 0.6 Gy/s, and 0.06 Gy/s) as those in Fig. 2. These lines divide the elongation versus gel-fraction planes into various sections. The distribution of other data points (expressed as letters) within these sections corresponds to their dose and dose rate almost without exception. This indicates that at the same doses and dose rates the damaging effect depends on the absorbed dose and not on the source or the radiation type. It should be especially noted that the data points of the CERN SPS pulsed irradiation, with its complex composition (γ , μ , p, n, etc.), are appropriately located in these figures within the experimental error.

Although the dose rate of the irradiation was calculated from the absorbed dose divided by the operation time of the accelerator, the situation is entirely different from that of continuous irradiations. The accelerator supplies radiation pulses of a few milliseconds down to 10^{-5} s (according to the mode of operation) about every 12 s. Therefore, the instantaneous dose rate must be much higher than the apparent calculated dose rate. There also exists an interval of about 10 s when there is no radiation between the pulses. If within this interval the oxygen diffusion were fast enough to restore a sufficient quantity of oxygen for the oxidation reaction of polymer radicals, the oxidation scission would predominate throughout the irradiated material. On the contrary, if the oxygen diffusion were much slower, the material would be occupied by a cross-linking region. However, the gel-fraction values of the SPS irradiations are between those of the ^{60}Co [(e), (f)] and the spent-fuel-element [(g), (h), (i)] irradiations. Also, the dose rates averaged over the operation time of the accelerator are between these two irradiations. This result therefore indicates that the CERN SPS irradiation may be treated as a continuous one.

3.2.2 Amino-type stabilized compounds (Materials 523, 529, and 530)

The two thermoplastic EVA sheathing materials 529 and 530 have the same fire protection and differ only by an addition of EPR (529) (see Fig. 4a) and an ozone protection (530). Their response to

ionizing radiation can be considered identical. As a similar performance is obtained with LDPE 523 (Fig. 4b), it is assumed that this is due to the amino-type stabilizer which is present in all three materials. This is also demonstrated by comparison of the elongation-at-break results with the gel-fraction measurements, of which Fig. 5 is a typical example. At 0.5 MGy and dose rates below 60 mGy/s there is less mechanical damage than at higher dose rates. These results show that damage caused by long-term irradiations at low dose rates is not necessarily more severe than damage resulting from short-term tests at high dose rates. The degradation depends on the effectiveness of the stabilizer and on the type of radiation degradation mechanism in the polymer.

3.2.3 Chemically cross-linked rubber compounds (Materials 531 to 535)

All materials of this group show a low dependence on dose rate and radiation type. This is because they were all cross-linked and stabilized at the time of fabrication. The cross-linked structure was not affected by irradiation, as shown by the results of gel-fraction measurements in Fig. 6. This means that the scission which is typical at low dose rates does not destroy the basic cross-linked network to any significant extent even though it causes the decrease of the cross-linking density, as is suggested by the higher measured values of the swelling ratio at lower dose rates.

Comparing the mechanical properties of the EPR insulating and sheathing materials (Figs. 7a and 7b), it can be noted that, apart from the small dose-rate effect, the residual elongation at the high doses is the same despite the different initial values. Figure 7c shows an example of a cross-linked EVA insulation, where again only minor differences can be observed in the results from the various radiation sources. Figure 7d gives a comparison of the EPR, EVA, and SIR materials irradiated at the CERN SPS. The results for the other four radiation sources are similar. Also, a stronger degradation for SIR is observed at higher doses, compared with the other elastomers.

3.3 Dose-rate dependence of end-point doses

The end-point doses can be defined by either interpolation as 50% of initial value, or as 100% absolute value of elongation at break. Of these two criteria, the 100% levels are always situated at higher doses and closer to the data points; therefore this criterion is used for the presentation of the end-point doses as a function of dose rate in Fig. 8.

Figure 8a, for the LDPE materials, clearly demonstrates that the different amount of stabilizers becomes important only at dose rates below 0.5 Gy/s, where the end-point doses systematically increase when more stabilizer has been added. Considering that these results at low dose rates have been obtained at three very different radiation sources, the consistent ordering by stabilizer content in Fig. 8a supports the assumption that radiation damage depends on absorbed dose and not on the type of radiation or energy distribution, and that for the present experiment different quality factors need not be applied.

The dependence of end-point doses on dose rates for EPR, SIR, and EVA compounds is shown in Fig. 8b for the same end-point criteria as above. Generally, a systematic relation between end-point dose and dose rate has not been observed. The dependence on dose rate is much less pronounced in the above materials than in LDPE, which can readily also be seen from Fig. 7 in Section 3.2.3. The effect of low dose rates for the EPR, SIR, and EVA materials is rather small and partly within the measurement errors.

3.4 End-point dose extrapolation to lower dose rates for LDPEs

In the preceding sections it has been clearly demonstrated that dose-rate effects are particularly pronounced in LDPE compounds. On the other hand, irradiations at low dose rates are very time-consuming, and extrapolation from experiments at higher dose rates is required. A possible approach

for dose rates below 60 mGy/s is demonstrated in Fig. 8a, where the straight lines correspond to a proportionality of end-point doses and the n^{th} power of dose rates, with $n = 0.5 \pm 0.2$. This result has been expected under the assumption that a peroxy chain-reaction degradation mechanism is fully operative. In the discussion of a previous long-term irradiation experiment [3] a relation between the end-point dose D_e and dose rate $\dot{D}$ has been used in the limit of small dose rates [8, 9]:

$$D_e \approx \sqrt{(k_3 g \dot{D}) / \{k_2 [R] + \sqrt{(k_3 g \dot{D})}\}} .$$

Here, g is the generation constant of radiation-induced radicals, $[R]$ the concentration of polymer chains available for production of radicals (assumed to be constant); k_2 and k_3 are the rate constants for the peroxy-induced radical production and termination of the peroxy chain reaction. Thus, if $\dot{D}$ is small and the number of new radicals not involved in the peroxy chain reaction can be neglected against the square of the number of radicals produced by this reaction, the end-point dose becomes proportional to the square root of dose rate. In the opposite case, the end-point doses do not depend on dose rates, as the two square roots in the above relation cancel out. Therefore, depending on how well the oxidative degradation is blocked by additives or by the chemical properties of the polymer itself, the end-point doses are expected to be proportional to a power n of dose rates within about $0 < n < 0.5$.

Of course, this simple relation could hold only at low enough dose rates, where the degradation is dominated by the reactions considered in its derivation [8, 9]. For instance, for oxygen to be available throughout a sample of our type and thickness, dose rates should be lower than 30–60 mGy/s. Between 0.6 and 7 Gy/s, degradation of mechanical properties due to cross-linking becomes dominant over oxidative degradation, as can be seen from our results. Therefore, if it were necessary to extrapolate to conditions of dose rates lower than those in this experiment, the square-root relation would be applied, starting from measured values preferably at dose rates below 0.1 Gy/s. At extremely low dose rates, the exposure times for reaching the end-point dose are very long, and other degradation mechanisms could become more important. Also, differences due to stabilizers could show up in irradiations over many years, although nothing has been detected in this experiment. Nevertheless, on the basis of the results presented, a selection of materials for an application using irradiations at a dose rate below 0.1 Gy/s offers the best safeguard against unexpected degradation due to dose-rate effects. Of course, life exposure over many years should be continued whenever possible [10], since the result of a test on a property as sensitive as elongation at break will give an indication of a mechanical breakdown early enough for action to be taken before such a failure actually occurs.

4. CONCLUSIONS

The results of the present study are an important contribution to our understanding with respect to predictions of radiation degradation of organic cable-insulating and cable-sheathing materials, in particular as in practice they have to be assessed from accelerated tests [11].

- 1) It has been confirmed that a few millimetres thick LDPE compounds are very sensitive to dose-rate effects and that stabilizers only become effective below 30 to 60 mGy/s. Therefore, important errors can be made—exceeding an order of magnitude—when assessing the radiation damage of these materials by means of accelerated tests.
- 2) Well-stabilized LDPEs and cross-linked rubber compounds—EPR, EVA, EAR, and SIR—do not show significant dose-rate effects within the 0.01 to 70 Gy/s covered by this experiment.
- 3) Taking the dose-rate effect into account, the end-point dose of 50% of the initial value of elongation at break would be obtained between 10^4 and 10^5 Gy for standard LDPEs, between 5×10^4 and 10^5 Gy for SIR, and between 10^5 and 5×10^5 Gy for cross-linked EPR, EVA, and EAR compounds.

- 4) From the interpretation of the elongation-at-break data, their correlation to gel-fraction measurements, and the interpretation of the influence of stabilizers on LDPEs, it can be concluded that the radiation damage is related to the absorbed dose irrespective of the different types of radiation used in this experiment.

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Table 1
Description of materials

Mat. No.	Polymer base	Type & applic.	Additives: type, weight (%)	Thickness (mm)
522	LDPE	TP, I	None	1.9
523	LDPE	TP, I	a, 0.4	2.1
524	LDPE	TP, I	tp, 0.08	2.0
525	LDPE	TP, I	p, 0.08	2.0
526	LDPE	TP, I	p, 0.15	2.0
527	LDPE	TP, I	p, 0.30	2.0
528	LDPE	TP, I	p, 0.15, OZ	2.0
529	EVA/EPR	TP, S	FR, 57	1.2
530	EVA	TP, S	FR, 57, OZ	1.2
531	EPR	XL, I	U, 70	1.5
532	SIR	XL, I		1.7
533	EVA	XL, I	U, 60	1.7
534	EVA	XL, S	FR, 65	1.8
535	EPR/EAR	XL, S	FR + U, 60	2.0

Explanations:

Polymer base:

LDPE : Low-density polyethylene, based on Lupolen 1810 D (BASF)

EVA : Ethylene vinyl acetate, Levapren (Bayer), Escorene (Esso)

EPR : Ethylene-propylene rubber Buna AP (Hüls), Keltan (DSM), Vistalon (Esso)

SIR : Silicone rubber, Silopren (Bayer)

EAR : Ethylene-acrylate rubber, Vamac (DuPont)

Type and application:

TP : Thermoplastic

XL : Cross-linked

I : Insulation

S : Sheath

Additives:

FR : Flame-retardant filler, alumina-trihydrate

U : Unspecified other fillers and additives

OZ : Ozone protection, ozone wax

a : Amino-type stabilizer, Hydroquinoline derivative

tp : Phenolic-type stabilizer, Diarylthioether derivative

p : Phenolic-type stabilizer, Pentaerythrite derivative

All samples cut from extruded material, except 529 and 530.

Materials 523, 524, 525, 529, and 535 are equally well stabilized against thermal degradation.

Table 2

Listing of radiation sources, doses, and dose rates. Each letter (a) to (s) represents one package of five tensile samples for each of the fourteen materials (522–535, see Table 1).

Radiation source	Radiation field	Sample series	Dose (kGy)	Dose rate (Gy/s)
Reactor ASTRA Ebene 1	$4 \times 10^{15} \text{ n}_{\text{th}}/\text{m}^2 \text{ s}$ $3 \times 10^{14} \text{ n}_{\text{f}}/\text{m}^2 \text{ s}$ $\approx 95\% \text{ gamma dose}$	a	500	70
		b	1000	56
		c	1900	7.8
		d	5000	56
^{60}Co source	Gamma rays 1.2 MeV	e	500	0.64
		f	1000	0.64
Spent-fuel elements	Gamma rays 0.5 to 3.0 MeV	g	220	0.061
		h	490	0.057
		i	1000	0.053
Synchrotron radiation at PETRA (DESY)	X-rays $E_{\text{crit}} \approx 60 \text{ keV}$ $E_{\text{eff}} \approx 100 \text{ keV}$	j	77 ± 33	0.008 ± 0.003
		k	–	–
		l	240 ± 88	0.023 ± 0.010
		m	2100 ± 370	0.06 ± 0.01
		n	53 ± 13	0.014 ± 0.005
CERN SPS targets	Hadron cascade and secondary gamma rays. Primary proton energy up to 450 GeV	o	525 ± 126	0.12 ± 0.03
		p	4000 ± 1000	0.47 ± 0.11
		q	1880 ± 300	0.62 ± 0.12
		r	5400 ± 1400	1.15 ± 0.25
		s	340 ± 65	0.052 ± 0.01

Figure captions

- Fig. 1** Elongation at break (in percent) as a function of the absorbed dose, for different types of LDPEs (see points in figures) at various radiation sources: a) reactor and ^{60}Co ; b) spent-fuel elements; c) PETRA (DESY); d) LDPE 525 for all sources used in this experiment (see points in figure).
- Fig. 2** Typical degradation behaviour of low-density polyethylenes, shown by correlation between the mechanical property degradation and the chemical property change. (Nos. 522, 524–528).
- Fig. 3** Correlation between the gel-fraction change and the degradation of elongation at break for LDPE 524 by irradiation. $\circ$ Reactor ASTRA; $\square$ ^{60}Co ; ∇ CERN SPS; $\triangle$ Spent-fuel element; $\diamond$ PETRA (DESY).
- Fig. 4** Elongation at break (in percent) as a function of the absorbed dose, at five different radiation sources (see points in figures) for a) EVA 529 and b) LDPE 523.
- Fig. 5** Correlation between the gel-fraction change and the degradation of elongation at break by irradiation for LDPE 523 containing an amino-type stabilizer. $\circ$ Reactor ASTRA; $\square$ ^{60}Co ; ∇ CERN SPS; $\triangle$ Spent-fuel element; $\diamond$ PETRA (DESY).
- Fig. 6** Correlation between the gel-fraction change and the degradation of elongation at break by irradiation for chemically cross-linked EVA 534. $\circ$ Reactor ASTRA; $\square$ ^{60}Co ; ∇ CERN SPS; $\triangle$ Spent-fuel element; $\diamond$ PETRA (DESY).
- Fig. 7** Elongation at break (in percent) as a function of the absorbed dose, at five different radiation sources (see points in figures) for a) EPR 531, b) EPR 535; c) EVA 533; d) is for rubber compounds irradiated at the CERN SPS (see points in figure).
- Fig. 8** End-point doses as a function of dose rates for the end-point criterion of 100% absolute value of elongation at break (see text): a) for LDPEs with various stabilizers and b) for various rubber compounds (see points in figures).

a)

b)

Fig. 1

c)

d)

Fig. 1

Fig. 2

Fig. 3

a)

b)

Fig. 4

Fig. 5

Fig. 6

Fig. 7

c)

d)

Fig. 7

a)

Fig. 8

b)

Fig. 8