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Determination of Dose Rate Effects in Polymers Irradiated in Vacuum

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The most reliable information on polymeric material radiation resistance and lifetime may be obtained in tests conducted under operational conditions. An important problem of the environment simulation is the adequacy of the accelerated test results, because the dose rates used in the test and under operational conditions can differ by up to several orders of magnitude. Accelerated testing to determine operational engineering performance is valid only when the change in a material property does not depend on the dose rate. It is well known that under irradiation in an air environment, the dose rate effects in polymers are very significant and are connected with oxidizing radiation destruction. For example, they are taken into account for polymer materials used in nuclear energy facilities. We examine an opposite situation, the irradiation of materials in vacuum that is typical for operation in space environments. It is often assumed that irreversible radiation changes in polymer properties do not depend on the dose rate. On the basis of available experimental data, including unpublished results obtained in our laboratory, we show that there are remarkable dose rate effects in physical–chemical and operational (e.g., mechanical, electrical, thermal, and optical) properties for a wide variety of polymers irradiated in vacuum. The material property change attributed to dose rate is often significant, varying by as much as an order of magnitude when compared to material response in an operational environment. In a number of cases, dose rate effects produce a nonlinear response in the material property.

Nomenclature

C^* = nonequilibrium state concentration
c = concentration of the crosslinks
D = absorbed dose, MGy
E = resulting radiolysis product concentration
F = surface area, m^2
G = radiation-chemical yield, 1/100 eV
I = dose rate, Gy/s
k = rate constant of chemical reaction, kJ/mol
k_D = diffusivity, m^2/s
l = sample characteristic size, m
M_n = number-averaged molecular weight
P = radiolysis product concentration
q = gel fraction
R = intermediate active species concentration
S = optical density
T_{ir} = temperature of irradiation
t = exposure time in seconds
W = product accumulation rate, s^{-1}
δ = dielectric losses
ε = elongation at rupture
λ = heat conductivity, $W/m \cdot K$
σ = relative tensile strength, MPa
τ = radiolysis species lifetime, s

Subscripts

a = under accelerated conditions
g = gaseous products
o = under operating conditions
1, 2, 3 = order of chemical reaction

Introduction

IRREVERSIBLE dose rate effects in polymer properties under irradiation in an oxygen environment are very significant and are based on chain reactions of oxidizing radiation destruction initiated by radiolysis species of radical type.[1–5] It is usually assumed that under irradiation in vacuum, such effects are negligible.[2–6]

The dose rate effects in polymers under irradiation in air, and the absence of such effects under irradiation in vacuum, or inert environment, were pointed out at first by Charlesby.[7] Wilski[2] made similar conclusions mainly about mechanical properties of polymers. Later they were supported by the data for gel formation in low-density polyethylene (LDPE) and ethylene–propylene copolymer irradiated in vacuum in the dose rate range from 0.26 to 2.8 Gy/s (Ref. 8). Similar results are obtained for gas evolution, gel formation, and mechanical properties of LDPE, polypropylene (PP), silicone, fluorine, and natural and isobutylene–isoprene rubbers in the dose rate range from 2.6 Gy/s for gamma radiation to 130 Gy/s for electrons.[9] No significant effects were observed for discoloration of cellulose triacetate or radiation scission of polymethyl methacrylate (PMMA) and polycarbonate (PC) under irradiation in the absence of oxygen at room temperature in the dose rate range from 4×10^{10} Gy/s for pulse electron radiation to 0.5 Gy/s for gamma radiation.[10]

The observed dose rate dependencies are sometimes based not on the specificity of the radiation-chemical processes, but on the influence of the elevated irradiation temperature at high dose rates. For example, a significant increase in Kapton optical density was discovered when 1-MeV proton flux density increased to 2×10^{13} (particles/cm^2 s).[10,11] Our calculations indicated a very high irradiation temperature for this proton flux.

When the dose rate dependence of the radiation effects on polymers in an oxygen-free environment can be neglected, both the International Electrotechnical Commission standard[12] and the National Russian Standard[13] limit the acceleration rate of polymer material radiation tests by the control of irradiation temperature. These

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limits for space materials operating in high-vacuum environments are not sufficient because they relate, as a rule, to dose rate values $I > 0.1$ Gy/s.

This distinction does not permit extrapolation to the range of $10^{-5} < I < 10^{-2}$ Gy/s, which is typical for space dose rates in low earth orbits. That is why the ESA standard recommends providing preliminary testing for each space material and each property and finding possible dose rate effects.[14]

Experimental Data

We now have sufficient experimental data demonstrating the dose rate dependence of radiation effects in vacuum. The main requirements for inclusion of the data presented below are availability of the conditions of experiment, irradiation, and measurement in the original source and information about the irradiation temperature T_{ir}. Dependency of gas evolution, optical spectra, radiation crosslinking, and radical generation are presented in Figs. 1–8. The high-dose-rate data are presented in Figs. 3–8. The data shown in Figs. 3 and 5 were obtained during irradiation at 293 and 296 K in an air environment,[15–17] and at 298 K in a nitrogen environment.[18] These values of T_{ir} were carefully controlled by gaseous nitrogen cooling.[19] These data are shown in Figs. 4 and 7. The same process for controlling T_{ir} has been utilized previously.[20,21]

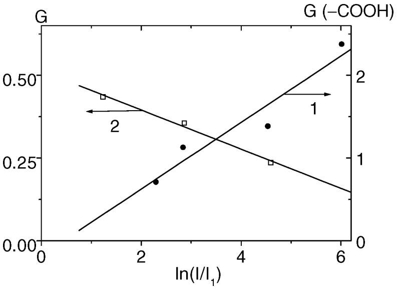

Fig. 1 Radiation-chemical yield of radiolysis species in mol/100 eV vs gamma-radiation dose rate in polyethyleneterephtalate (PETP). Curve 1 represents the carboxylic groups; curve 2 represents the gas species.[15,16]

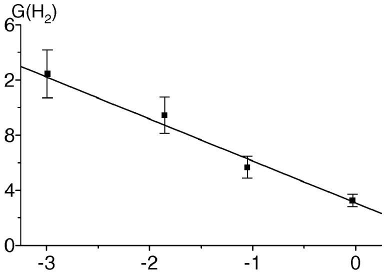

Fig. 2 Hydrogen radiation-chemical yield in mol/100 eV vs gamma-radiation dose rate in the material LDPE.[15,16]

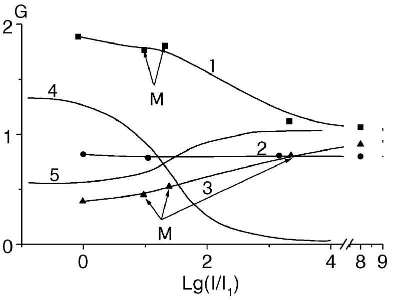

Fig. 3 Radiation-chemical yield of radiolysis species vs gamma-radiation dose rate. Radiolysis of polydimethylsiloxane in mol/100 eV is shown in curves 1–3 and of neopentane is shown in curves 4 and 5.[17] Curve 1 shows the methane species, curve 2 shows the hydrogen, curves 3 and 5 show ethane, and curve 4 shows the 2,2,5,5-tetramethylhexane species. The points, for polydimethylsiloxane marked by "M" were obtained from Miller.[18]

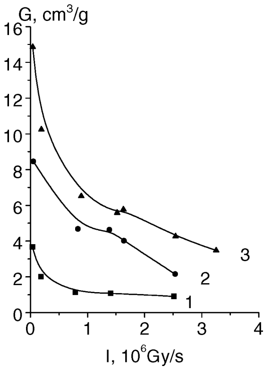

Fig. 4 Concentration of total radiolysis gas species in PP vs dose rate at doses of 1, 1 MGy; 2, 2.5 MGy; and 3, 5 ÌGy of 5-MeV electron radiation at room temperature.[19]

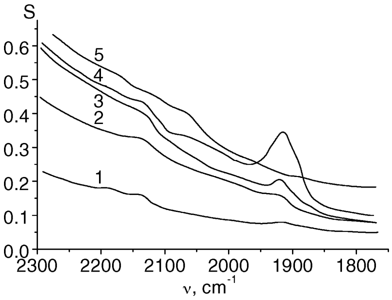

Fig. 5 Absorption spectrum of irradiated PS, shown in curves 1–4, and polyvinylxylene, shown in curve 5. Dose rates used during irradiation were 1, 0.01; 2 and 5, 0.17; 3, 2.15; and 4, 4 × 10^5 Gy/s (Refs. 16 and 17).

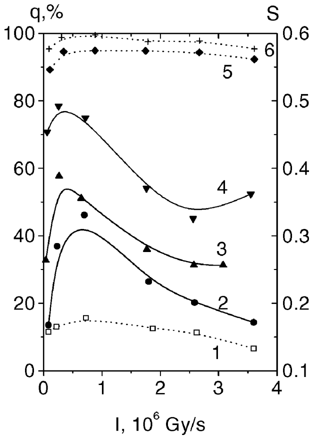

Fig. 6 LDPE gel fraction *q* shown in curves 1, 5, and 6 and optical density *S* at 970 cm^{-1} shown in curves 2–4 vs dose rate. LDPE was irradiated with 5-MeV electron radiation to absorbed dose levels of 0.02 MGy for curve 1, 1.2 MGy for curves 2 and 5, 2.5 MGy for curve 3, and 5 MGy for curves 4 and 6.[20]

All data for high dose rates, more than 10^3 Gy/s, were obtained under pulsed electron irradiation. The maximum values of the single-pulse dose rate are indicated in Figs. 4 and 6–8. Taking into account the pulse frequency and the duration of a single pulse, the average value of the dose rate for the pulse train is about 10^3 times lower. Figures 3 and 8 indicate that high values of dose rate (i.e., >100 Gy/s) will not noticeably change the character of the experimental curves. In any case, the pulse mode of irradiation possesses its own features of polymer material radiolysis and is not an object of our investigation.

If irradiation at high dose rates is accompanied by significant increase of temperature, the radiation effects are to be increased in accordance with the Arrhenius approximation. The realistic application of the high-dose-rate situation is, as a rule, quite different. During the irradiation the average temperature of the material samples cannot exceed the specified temperature level. The dependence of radiation-chemical yields on dose rate is presented in Figs. 1–3.[15,16] In these figures, the value of I_1 is equal to 1 Gy/s.

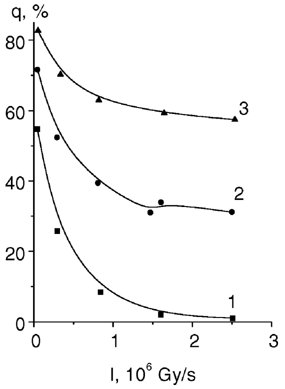

Fig. 7 **Gel fraction, q, in PP vs dose rate at dose levels of 1, 1; 2, 2.5; and 3, 5 MGy (Ref. 19).**

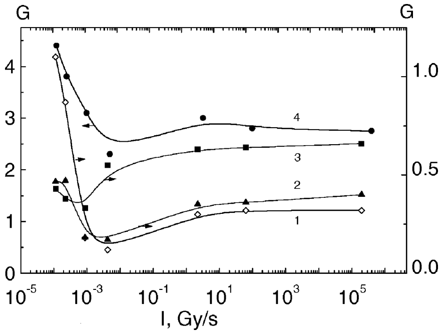

Fig. 8 **Radiation-chemical yields of paramagnetic centers, G, 1/100 eV, vs dose rate.**

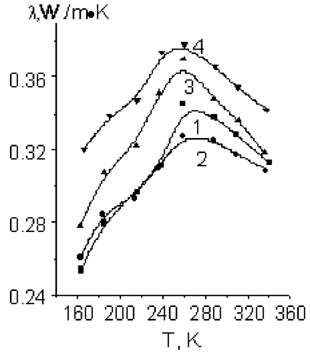

Fig. 9 **LDPE heat conductivity vs temperature. The total absorbed dose was 1 MGy. Curve 1 shows data from the unirradiated sample, curve 2 shows data from LDPE irradiated at a rate of 1 Gy/s, curve 3 shows data from LDPE irradiated at a rate of 10^{-1} Gy/s, and curve 4 shows data from LDPE irradiated at a rate of 10^{-2} Gy/s (Refs. 22 and 23).**

Table 1 **Relative tensile strength σ/σ_0 and elongation at rupture $\varepsilon/\varepsilon_0$ vs dose rate for PTFE and LDPE**

	PTFE at dose 3 kGy		PTFE at dose 100 kGy	LDPE at dose 300 kGy	
I, mGy/s	σ/σ_0	$\varepsilon/\varepsilon_0$	σ/σ_0	σ/σ_0	$\varepsilon/\varepsilon_0$
0.9	0.5	0.3	——	1.4	0.8
14.5	0.5	0.6	0.3	1.3	0.8
85	——	——	0.5	1.3	0.9
910	0.7	0.9	0.7	1.1	1.0

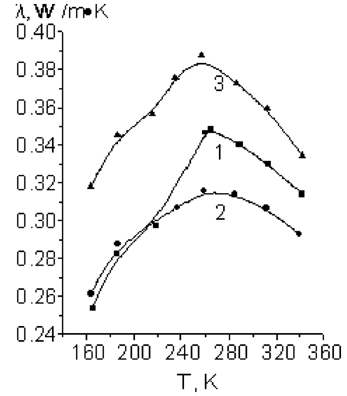

Fig. 10 **LDPE heat conductivity λ vs temperature. The total absorbed dose was 10 kGy. Curve 1 shows data from the unirradiated sample, curve 2 shows data from LDPE irradiated at a rate of 10^{-2} Gy/s, and curve 3 shows data from LDPE irradiated at a rate of 10^{-3} Gy/s (Refs. 22 and 23).**

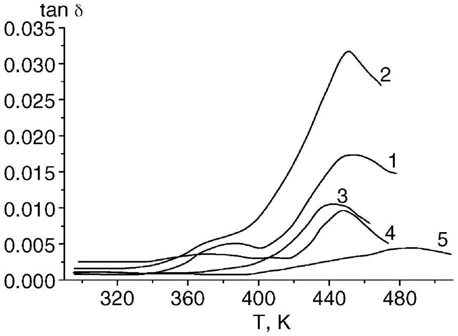

Fig. 11 **Tangent of dielectric losses, tan δ (frequency 1 kHz), vs temperature for PTFE irradiated in vacuum by 100 kGy for curves 1–3 and 30 kGy for curves 4 and 5. Dose rates for this exposure were 0.01 Gy/s for curves 1 and 4, 0.1 Gy/s for curves 2 and 5, and 1 Gy/s for curve 3 (Refs. 22 and 23).**

It can be seen that the radiation-chemical yield of gases changes when the dose rate increases. It is well known that the radiation-chemical yields increase with increasing temperature. Hence one can expect only an increase in the gas yield when the dose rate rises without thermosetting, in contrast with the data presented by Serenkov et al.[19] The dependence of the gel fraction q and the optical density S on dose rate is presented in Figs. 5–7.

Figure 8 shows the data for the dependence of radical generation on dose rate. The polystyrene (PS) is represented by curve 1, PC by curve 2, sodium–silicone glass by curve 3, and PMMA by curve 4. Irradiation occurred in vacuum at room temperature.

Radiation-induced changes in the mechanical, thermal, and electrical properties of polytetrafluoroethylene (PTFE) and LDPE upon irradiation in vacuum in the dose rate range of 10^{-3}–1 Gy/s are presented in Table 1. Figures 9–11[22,23] graphically show these radiation-induced changes in LDPE and PTFE.

Data on tensile strength of PP irradiated up to 2.5 MGy are presented by Serenkov et al.[19] Different rates of ethylene–vinyl acetate copolymer degradation (i.e., gel fraction, gas evolution, swelling, strength) under gamma radiation and electron impact with varying dose rates were observed by Haruyama et al.[9] The small difference in high-density polyethylene gel fraction values at high dose rate, approximately 10%, agrees well with the data for LDPE presented in Fig. 6.[10,20] The dose rate effects in operational material properties sometimes are not so large as for radiolytic species and the products of the chemical stage of radiolysis (e.g., crosslinking and scission). This is a result of gradual progression of the effect along the path from the physical stage of radiolysis via physical–chemical effects to the eventual degradation of the materials' operational properties. The same situation takes place in the process of linear energy transfer.

Dose Rate Effect Models

Theoretical prerequisites for the dose rate effects described are quite clear. A combination of radiolysis and the processes of molecular motion in polymers results in a change of the molecular and macromolecular structures of the material. The issue is only the extent of the effect. An indirect proof of the effect inevitability is the temperature dependence of the radiation degradation of the material when the number ratio of thermal activation events of molecular motion to radiation events of excitation and ionization changes.[24]

The physical reasons for dose rate effects on irreversible changes of polymer properties under irradiation in vacuum are the following: radiolysis product interaction; radiolysis product mass transfer; and relaxation character of molecular mobility. Let us examine each reason separately.

Radiolysis Product Interaction

Change of the polymer material properties results from a number of chemical and physical processes where particles and structures with different life, or relaxation time, take part. In principle, when the result of radiation impact depends on the interaction of two radiolysis products, including interaction with secondary radiolysis products, a dose rate effect will occur. Such a possibility was reported by Makhlis et al.[6] Kudoh et al.[10] describe a "threshold dose rate" for spur overlapping. This model indicates that increasing intermediate-radiolysis-product interaction will produce dose rate effects. But up to $I = 4 \times 10^{10}$ Gy/s such dependence was not discovered.

Let us examine the following generalized situation. Let the radiolysis product E be a result of the interaction of two radiolysis products P_1 and P_2. Then the accumulation kinetics will be described by the set of equations

$$\frac{\mathrm{d}[E]}{\mathrm{d}t} = k[P_1] \cdot [P_2] \tag{1}$$

$$\frac{\mathrm{d}[P_1]}{\mathrm{d}t} = G_1 I - k[P_1] \cdot [P_2] - \frac{[P_1]}{\tau_1} \tag{2}$$

$$\frac{\mathrm{d}[P_2]}{\mathrm{d}t} = G_2 I - k[P_1] \cdot [P_2] - \frac{[P_2]}{\tau_2} \tag{3}$$

where I is dose rate, G_1 and G_2 are the radiation-chemical yields of species P_1 and P_2, and τ_1 and τ_2 are radiolysis species lifetimes, which are determined as a result of all other decay processes. This set of nonlinear equations, as a rule, cannot be solved exactly. For simplicity, we shall examine some extreme cases.

Let us examine a stationary case and assume that the stationary concentration of the radiolysis species is mainly determined by destruction reactions (i.e., loss, escape from a reaction cell, etc.). Then

$$[P_1] = G_1 I \tau_1, \qquad [P_2] = G_2 I \tau_2 \tag{4}$$

$$\frac{\mathrm{d}[E]}{\mathrm{d}t} = k G_1 G_2 I^2 \tau_1 \tau_2 \tag{5}$$

It is clear that the accumulation rate of the product E depends quadratically on the dose rate. In another limiting case where the radiolysis species vanish only through decay processes, without interaction, we observe a linear dependence on the dose rate.

A material's dose rate dependency can be influenced by the interaction of radicals and excited states. Bol'bit et al.[16] discuss this dose rate dependence on radical yield. Also, a dose rate influence on the magnitude of the irreversible radiation effects may reveal itself when the radiolysis species are formed as a result of at least two processes of different kinetic order. For example, let the products P_1 and P_2 be formed through reactions of intermediate species R of first and second order. Then the process kinetics is described by the set of equations

$$\frac{\mathrm{d}[R]}{\mathrm{d}t} = GI - k_1[R] - k_2[R]^2 \tag{6}$$

$$\frac{\mathrm{d}[P_1]}{\mathrm{d}t} = k_1[R] \tag{7}$$

$$\frac{\mathrm{d}[P_2]}{\mathrm{d}t} = k_2[R]^2 \tag{8}$$

where R, P_1, and P_2 are intermediate active species and reaction products concentration, respectively. The radiation yield of the intermediate active particles is given by G, and the rate constants of the first- and second-order reactions are k_1 and k_2. The ratio of the accumulation rates of products P_2 and P_1 is described by the expression

$$W(P_2)/W(P_1) = \left[(1 + k_2 GI/k_1)^{0.5} - 1\right](k_2 GI)^{0.5}\Big/k_1 \tag{9}$$

So the ratio of product accumulation rates depends on the dose rate.

As a rule, several reactions involving intermediate active species occur simultaneously. In general, these reactions can be described as recombination reactions of second order and reactions with other molecules. A typical example is the methyl radical CH_3 that is formed by C–C bond scission in branched hydrocarbons. Ethane is formed as a result of methyl radical second-order recombination. Methyl radicals interact with hydrocarbon molecules through a first-order reaction and produce methane. The ratio of methane to ethane concentration depends on the dose rate. The dose rate range where this dependence is significant is determined by the ratio of constants k_1 and k_2. For example, the composition of gaseous radiolysis products of polysiloxanes and branched hydrocarbons is described by the expression of the type given above in the dose rate range 0.1–100 Gy/s.

Crosslinking is a result of macroradical recombination. In this case we can write the expression

$$\frac{\mathrm{d}c}{\mathrm{d}t} = k[R]^2 \tag{10}$$

$$\frac{\mathrm{d}[R]}{\mathrm{d}t} = GI - k[R]^2 \tag{11}$$

where c is concentration of the crosslinks. If the radicals are consumed only in crosslinking then the radiation bond yield is directly proportional to the dose rate.

However, the radicals can participate in other processes, including other radiolysis species. This process can be described as

$$\frac{\mathrm{d}c}{\mathrm{d}t} = k_1[R]^2 \tag{12}$$

$$\frac{\mathrm{d}[R]}{\mathrm{d}t} = GI - k_1[R]^2 - k_2[R]G \tag{13}$$

$$\frac{\mathrm{d}G}{\mathrm{d}t} = G_g I - k_2[R]G - k_3 F k_D G \tag{14}$$

where the third term of the last equation takes into account the gas evolution from the polymer volume. Solution for a stationary mode is given by a nonlinear dose rate dependence of the bond yield.

Radiolysis Species Mass Transfer

A significant amount of gaseous products in organic materials can be generated as a result of irradiation. They can take part in chemical reactions and diffuse from the polymer volume. Analysis of the kinetic equations describing these processes shows that the following limiting cases, determined by the ratio of diffusion time ($\tau \sim l^2/6\,k_D$) to exposure time ($t = D/I$), can be realized. If $\tau \ll t$, then dose rate effects may be neglected, because gases do not influence the radiation-chemical processes; they have time to leave the sample volume. In the opposite case, the dose rate effect will be apparent. The gaseous radiolysis products can influence the radiation effects on different polymer properties, particularly crosslinking and scission. For example, hydrogen and methane accelerate macroradical destruction, using a relay-race-type transfer of free valence. It is important that macroradical destruction may result both in macromolecule crosslinking because of recombination and in double bond formation by macroradical disproportionation. In the presence of hydrogen, the disproportionation reaction is more effective. This means that in these cases we can observe different crosslinking effectiveness with corresponding change in operational characteristics.

Mass transfer processes can also result in dose rate effects when the gaseous radiolysis products do not take part in the chemical reactions. These products can leave the volume or accumulate as bubbles (clusters) at defect points of the polymer. In the letter case, the gas accumulation can result in integrity fracture, generation of cracks, etc. The process direction is defined by competition of micro- and macrodiffusion; rate of gas generation, which is proportional to the dose rate; nucleation center concentration; polymer strength; relaxation rate; radiolytic gas solubility in the polymer; and sample thickness.

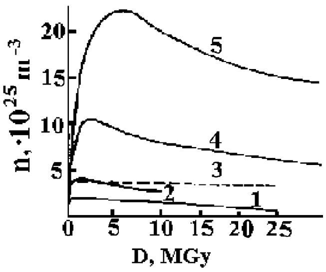

Fig. 12 Radiolytic gas concentration n in units of mol/m^3 vs absorbed dose D in LDPE at dose rates 1×10^2 Gy/s for curve 1, 2.7×10^2 Gy/s for curve 2, 6.4×10^2 Gy/s for curve 4, and 10×10^2 Gy/s for curve 5.

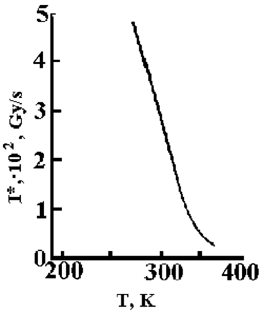

Fig. 13 Threshold dose rate value I^* vs irradiation temperature for LDPE.

The radiolytic-gas-concentration dependence on absorbed dose is presented in Fig. 12 for LDPE at irradiation temperature 293 K (Ref. 25). Dotted line 3 corresponds to the gas concentration when internal pressure in the free volume is equal to polymer strength at the specified temperature. The bubble generation begins after the curves intersect the straight line 3, that is, when the dose rate achieves some threshold value, I^*. Dependence of the threshold dose rate value, I^*, on the irradiation temperature for LDPE is presented in Fig. 13.

These effects were observed during investigation of reversible radiation effects on specific heat capacity of polymers and investigation of polydimethylsiloxane radiation cross-linking in vacuum at dose rates of 0.03 and 10 Gy/s (Refs. 25 and 26).

Relaxation Character of Molecular Mobility

Irradiation of polymer results in the formation of nonequilibrium states (i.e., excited states of molecules, radicals, ions, etc.), which have specific lifetimes that are often temperature dependent. The presence of nonequilibrium states first directly changes some properties of polymeric materials (i.e., optical, electrical, chemical) and second causes change of the properties through a process of relaxation. The magnitude of degradation depends on the degree of system nonequilibrium.

The rate of equilibrium upset is proportional to the dose rate:

$$\frac{dC^*}{dt} = GI - \frac{C^*}{\tau} \tag{15}$$

where G is the radiation-chemical yield of nonequilibrium states.

If it is assumed that G is independent of the dose rate, then a quasiequilibrium concentration of nonequilibrium states is established under irradiation:

$$C^* = GI\tau \tag{16}$$

It is defined by competition of generation and destruction. As to irreversible radiation effects, it is necessary to provide an equal concentration of nonequilibrium states during accelerated tests:

$$C_a^* = C_o^* \tag{17}$$

where indices a and o relate to accelerated and operating conditions. This means that as the dose rate rises, it is necessary to increase the relaxation rate, i.e., to decrease the relaxation time by

$$\tau_a = \tau_o(I_o/I_a) \tag{18}$$

The nonequilibrium states generated under irradiation are connected with the formation of intermediate active species that have differing natures, take part in different chemical processes, and have different relaxation times. For example, the lifetime of electron-excited molecules is about 10^{-7}–10^{-8} s, whereas lifetimes of charged particles vary over a broad interval. Lifetimes at low temperature can be as long as several hours. The lifetimes of macroradicals vary over a larger time range; some of them may even be stable at 300 K for several years. This means that during accelerated radiation tests, it is really impossible to reproduce the concentration of nonequilibrium states for different particles having different relaxation times and different activation energies of relaxation processes.

The following approximate approach is still possible based on the concentration of nonequilibrium states and their contribution to the radiation effect. It is necessary to compare the quantity of active species, C, generated during exposure and stationary concentration, C^*. If $C^* \ll C_o$, the relaxation processes may be neglected.

The above-described models are to be validated. It is very difficult to check them because 1) all of the mentioned mechanisms are acting simultaneously and 2) as a rule, the constants of radiation-chemical reactions are not known. We know only the test results for radiation effects on specific heat capacity of polyvinylformal and LDPE,[26] which validate the thermodynamic model of radiolytic gas expansion proposed by Briskman.[25]

Conclusions

A widespread opinion is that dose rate effects on polymer materials are observed only during irradiation in an oxygen environment. The observed effects under vacuum are usually ascribed to irradiation temperature influence. Our goal was to understand whether the dose rate effects on polymer properties under irradiation in vacuum really exist. Only in this case can we prove the reliability of accelerated radiation tests of materials operating in the space environment. With this end in view, we made a careful selection of all the available data, including our own earlier unpublished results. We presented only those data that do not result from the irradiation temperature variation. We also suggested also several simple models, not to calculate the dose rate effects but to show that these effects are inevitable. Our conclusions are as follows:

1) Radiation effects on polymer properties under irradiation in vacuum depend, in general, on the dose rate. This conclusion is contrary to previous prevailed opinion. The dose rate effect can reach an order of magnitude for intermediate radiolysis products. Along the path from the physical–chemical stage of radiolysis to the chemical stage, and then to the polymer properties, the magnitude of the effect significantly decreases.

2) Sometimes this dose rate dependence has an extremal character and may be a reason for observed discrepancies between different data.

3) Dose rate effects are observed mainly at long exposures and dose rates lower than 0.1 Gy/s.

4) The problem of dose rate effect in the radiation test may be solved by development of an adequate physical–chemical model or approximate simulation. But, as a rule, the development of such models requires extensive investigation. Moreover, such models are valid only within a relatively narrow range of variations in external parameters (e.g., temperature) defined, in particular, by phase or relaxation transition.

5) A method of approximate simulation is proposed for radiation tests of materials for space application. It consists of testing of representative material in the set of polymers for the dose rate interval defined by an acceleration factor. The method is recommended in the draft ISO standard "Space Environment Simulation at Radiation Tests of Materials, I. Nonmetallic Materials."

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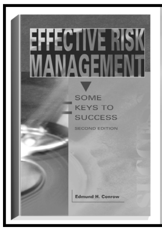

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