

The relationships obtained herein may be used to calculate true gas thermal conductivity over a wide temperature range from experimental data obtained by various methods.

NOTATION

U, energy; T, temperature; p, pressure; ρ , density; V, volume; v, specific volume; S, entropy; h, specific enthalpy; M, gas mass; m, molecular mass; L_{ij} , L_{ij} , phenomenological coefficients; λ , thermal conductivity coefficient; λ_{tr} , true thermal conductivity; λ_{ef} , effective thermal conductivity; D, self-diffusion coefficient; η , viscosity coefficient; c_v , specific heat at constant volume; k, Boltzmann's constant; θ^* , heat of transfer.

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AN ANALYTICAL MODEL FOR REVERSIBLE RADIATION EFFECTS ON POLYMER SPECIFIC HEAT

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A study is made of the energy balance in gas release by radiation in polymers as one of the possible mechanisms for reversible effects on the specific heat.

There are papers on irreversible radiation effects on the specific heats of polymers for polyethylene [1-3] and polystyrene [2, 4]. The effects are usually related to macrostructural transformations on irradiation and are examined when the radiation ceases.

Nothing is known about the reversible (instantaneous) effects on the specific heats of polymers. Moreover, it is not even clear whether such changes actually occur, although it is logical to assume that short-lived radiolysis products may affect individual modes in the collective vibrations (individual segments or the polymer chain as a whole) and can thus give rise to reversible effects.

We have examined one of the possible mechanisms for reversible change in the specific heat associated with the thermodynamics of gas production by radiolysis.

I. Theoretical Analysis. Although nearly all of the absorbed energy ultimately goes to heat the specimen, some is used in producing cross-linking chemical bonds, transvinylene unsaturation, etc. A certain fraction of the energy accumulates in the radiolytic gas in the free volume of the polymer.

If the relative rate of diffusion to the surface is sufficiently small, the gas pressure gradually increases and attains a certain critical value P_{cr} corresponding to the recrystallization stress σ_y (the yield point). From this time on, gas bubbles are formed, whose number and sizes gradually increase. This volume change occurs essentially at constant P_{cr} , since the stretching curve for an unoriented crystallized polymer is as shown in Fig. 1. Bubble growth corresponds to the part AB on the deformation curve.

The gas performs work $p dv$ in this isobaric process. There is correspondingly a change in the internal energy du . The total amount of heat dq supplied to the gas is

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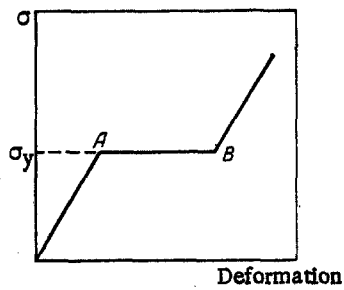


Fig. 1

Fig. 1. Dependence of mechanical stress on strain in an unoriented polymer.

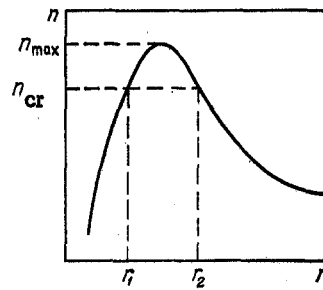


Fig. 2

Fig. 2. Relative dependence of radiolytic gas concentration on absorbed dose.

$$dq = du + pdv = di,$$

i.e., the heat consumption is equal to the enthalpy difference di for the expanding gas. It is more convenient to express dq in terms of pdv . Then

$$\alpha = \frac{du}{dq} = \frac{c_v d(T_2 - T_1)}{c_p d(T_2 - T_1)} = \frac{c_v}{c_p} = \frac{1}{k},$$

where $k = c_p/c_v$, so

$$dq = \alpha dq + pdv = \frac{k}{k-1} pdv. \quad (1)$$

In most cases at least, the main radiolysis products in a polymer produced by the δ electrons are diatomic gases. For example, the relative yield of radiolytic hydrogen for polyethylene is about 95%. As $k = 1.4$ for a diatomic gas,

$$dq = 3.5 pdv. \quad (2)$$

Therefore, to determine the instantaneous effects on the specific heat we have to relate P_{cr} and the gas volume to the absorbed dose and the dose rate, as well as the irradiation temperature. The subsequent calculations are for low-density polyethylene.

Determination of P_{cr} . A model has been devised [5] for the production of gas by radiation in polyethylene. In particular, analytic expressions have been derived for the gas molecule concentration by solving a system of differential equations for the diffusion and gas formation in the breakage of C-H bonds with allowance for the constants of the forward and reverse reactions. The formation of new C-C bonds leads to the production of gas molecules, and this is incorporated for three basic types of radiation-chemical reaction: 1) cross-linking bonds in the amorphous phase, 2) cross-linking in the crystalline phase, and 3) formation of transvinylene unsaturation.

The following equation was derived in [5] for the gas concentration n in mole/m³:

$$n = I \sum_{i=1}^3 \frac{K_{i1}}{1/\tau_D - IK_{i2}} [\exp(-IK_{i2}\tau) - \exp(-\tau/\tau_D)]. \quad (3)$$

The value of n at which bubble growth occurs satisfies

$$n = SP_{cr} = S\sigma_y, \quad (4)$$

where S is the solubility constant for the radiolytic gas (hydrogen). We substitute (4) into (3) to get

$$S\sigma_y = I \sum_{i=1}^3 \frac{K_{i1}}{1/\tau_D - IK_{i2}} [\exp(-IK_{i2}\tau) - \exp(-\tau/\tau_D)]. \quad (5)$$

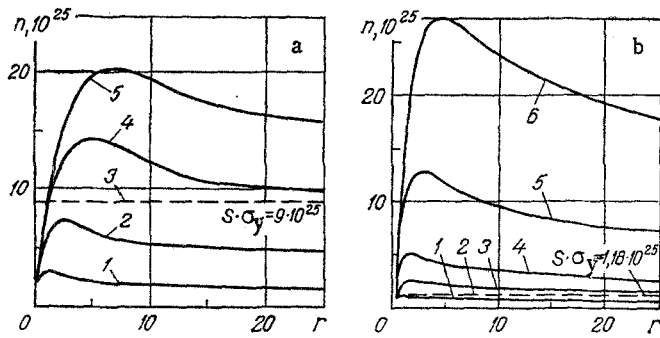


Fig. 3

Fig. 3. Dependence of radiolytic-gas concentration on absorbed dose for polyethylene. The parameter is the absorbed dose rate I : a) irradiation temperature $T = 293^\circ\text{K}$, 1) $I = 1 \cdot 10^2$; 2) $2.7 \cdot 10^2$; 4) $6.4 \cdot 10^2$; 5) $10 \cdot 10^2$; b) $T = 373^\circ\text{K}$, 1) $I = 0.2 \cdot 10^2$; 3) $0.5 \cdot 10^2$; 4) $1 \cdot 10^2$; 5) $2.7 \cdot 10^2$; 6) $6.4 \cdot 10^2$. I , Gy/sec; n , $1/\text{m}^3$; r , MGy; S , m^{-3} .

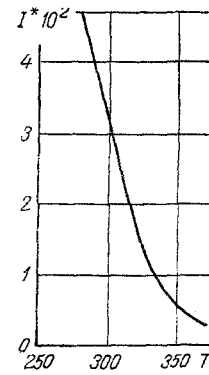


Fig. 4

Fig. 4. Dependence of threshold absorbed dose rate I^* on irradiation temperature T in $^\circ\text{K}$.

For given S and σ_y we use the absorbed dose rate I as parameter, and then we can derive the absorbed doses $r = Ir$ corresponding to the start of bubble formation. Since the characteristics of the material (S , σ_y , and τ_p) are dependent on temperature, as are the reaction constants, r will be a function of irradiation temperature.

Determination of v . Here there are two possible approaches: a purely analytic one based on examining the gas-production parameters and calculation of the total bubble volume from experimental data on the microstructures of the irradiated specimens.

Calculation of v from Gas-Formation Parameters. The total number N of gas molecules produced up to time τ is

$$N = \sum_{i=1}^3 \int_0^{\tau} v_i(\tau) d\tau, \quad (6)$$

where $v(\tau)$ is the gas formation rate. According to [5]:

$$v_i(\tau) = v_{0i} \exp(-I\tau/a_i), \quad (7)$$

where v_{0i} is the gas production rate in reaction of type i for $\tau = 0$ and a_i is the absorbed dose for gas formation relaxation. We substitute (7) into (6) and integrate to get

$$N = \sum_{i=1}^3 v_{0i} \tau_{vi} [1 - \exp(-r/a_i)], \quad (8)$$

where τ_{vi} is the gas deposition relaxation constant and $\tau_{vi} = 1/(IK_{i2})$.

We consider the radiolytic gas as ideal to get the volume concentration v for pressure $P_{Cr} = \sigma_y$ and the corresponding temperature T :

$$v = 3.7 \cdot 10^{-26} \frac{P_0 T}{T_0 \sigma_T} \sum_{i=1}^3 v_{0i} \tau_{vi} [1 - \exp(-r/a_i)], \quad (9)$$

where P_0 and T_0 are the pressure and temperature under normal conditions: $P_0 = 10^5$ Pa, $T_0 = 293^\circ\text{K}$.

This value of v is the maximum one, since it includes the gas in the bubbles and in the free volume of the polymer formed throughout the irradiation time.

Clearly, the $n = f(r)$ curve of (3) has a maximum, which is due to the exponential fall in the gas formation rate in (7) and the increase in diffusion as the radiolysis product

TABLE 1. Values of the Upper Limiting Dose r_2 in MGy

T, K	$I, 10^2 \text{ Gy/sec}$				
	0,5	1,0	2,7	6,4	10
293	—	—	—	45	150
333	—	4	100	210	—
373	37	100	150	—	—

TABLE 2. Reversible Changes in Specific Heat ϵ'_c in % (Upper Bound)

$r, \text{ MGy}$	$T=333 \text{ K},$ $I=100 \text{ Gy/sec}$	$r, \text{ MGy}$	$T=373 \text{ K}$	
			$I=100 \text{ Gy/sec}$	$I=1000 \text{ Gy/sec}$
0,5	0,25	0,05	—	0,76
1	0,24	0,1	0,47	—
2	0,22	50	0,17	—
3	0,21	100	0,10	0,15
4	0,20	200	—	0,05

Note: The first dose value in each column corresponds to r_1 and the last to r_2 .

concentration increases. Figure 2 shows the relative form of the $n = f(r)$ curve. Bubbles are formed for $n_{\max} > n_{\text{cr}}$, where n_{cr} corresponds to P_{cr} . If $n < n_{\text{cr}}$, after some time, new bubbles will not be formed and some of the old ones may collapse. Therefore, (5) either has two solutions ($n_{\max} > n_{\text{cr}}$) r_1 and r_2 (r_1 is the dose for the start of bubble formation and r_2 is the dose for completion of bubble formation) or else one solution ($n_{\max} = n_{\text{cr}}$), or none at all ($n_{\max} < n_{\text{cr}}$).

We are interested in the case where $n_{\max} > n_{\text{cr}}$. Two limiting estimates can be made for v : an upper bound in that beginning with r_1 all the gas formed diffuses to bubbles. Then the integration in (6) is from r_1 to r ($r_1 \leq r \leq r_2$):

$$v' = 3,7 \cdot 10^{-26} \frac{P_0 T}{T_0 \sigma_y} \sum_{i=1}^3 v_{0i} \tau_{vi} [\exp(-r_1/a_i) - \exp(-r/a_i)]. \quad (10)$$

The lower bound is when part of the gas diffuses to the surface. Then from (3) we have

$$v' = 3,7 \cdot 10^{-26} \frac{P_0 T}{T_0 \sigma_y} \left\{ I \sum_{i=1}^3 \frac{K_{i1}}{1/\tau_D - IK_{i2}} [\exp(-r_1 K_{i2}) - \exp(-r/I\tau_D)] - S\sigma_y \right\}, \quad (11)$$

$$r_1 \leq r \leq r_2.$$

Calculation from Microstructure Data. Unfortunately, only very restricted evidence is available on the volumes of the pores and cracks formed in polymers on irradiation. In [6] it was found that on irradiation of polyethylene at 77°K at doses of 20, 45, and 62 MGy, 1 cm³ of polymer retains about 100, 260, and 285 cm³ of hydrogen correspondingly, i.e., the gas pressure was about 30 MPa. In an unfrozen specimen at an external pressure of 15 MPa, the saturation concentration is only 3.8 cm³ of hydrogen per cm³.

The microstructure has been examined for low-density polyethylene irradiated up to 1 MGy [7] in nitrogen, and it was found that the numerical pore concentration as a function of dose had a maximum in the region of 0.1 MGy at 32 pores/ μm^2 . The average pore size was $0.100 \pm 0.025 \mu\text{m}$. However, the authors of [7] were not convinced that these pores were due to irradiation rather than the freezing in liquid nitrogen for electron microscopy. For this pore size and concentration, the surface concentration ρ_s was 0.24. According to [5], irradiation with reactor radiation at 2.4 kGy/sec gave $\rho_s = 0.16$ (our estimate from a photograph of the section with allowance only for the large pores). To determine v , one needs to transfer to the bulk concentration ρ_v . We represent the pores as spheres with a uniform distribution in volume to get

$$\rho_v = \frac{4}{3V\pi} \rho_s^{3/2} \approx 0.75 \rho_s^{3/2}. \quad (12)$$

These values of ρ_V are used below in numerical calculations.

Determination of Reversible Effects on the Specific Heat. We determine the reversible effects as the ratio of the amount Q of heat supplied to the gas during expansion to the amount R of radiation energy absorbed by the specimen. In accordance with (2)

$$Q = \int dq = 3.5 P_{cr} \int_{r_1}^r dv = 3.5 P_{cr} v, \quad R = (r - r_1) \rho.$$

In accordance with (10) and (11), the upper and lower bounds for Q are

$$Q' = 3.5 P_{cr} v', \quad Q'' = 3.5 P_{cr} v''.$$

One can calculate two values for the specific-heat change: $\bar{\epsilon}_c$, the mean integral change within limits from r_1 to r , and ϵ_c , the instantaneous change in specific heat for $r_1 \leq r \leq r_2$:

$$\bar{\epsilon}_c = Q/R; \quad \epsilon_c = \frac{\partial Q / \partial \tau}{\partial R / \partial \tau}; \quad \partial R / \partial \tau = I \rho.$$

We get correspondingly for the upper and lower bounds ϵ' and ϵ'' that

$$\bar{\epsilon}'_c = \frac{1.3 \cdot 10^{-25} \frac{P_0 T}{T_0} \sum_{i=1}^3 v_{0i} \tau_{vi} [\exp(-r_1/a_i) - \exp(-r/a_i)]}{(r - r_1) \rho}; \quad (13)$$

$$\epsilon'_c = 1.3 \cdot 10^{-25} \frac{P_0 T}{I \rho T_0} \sum v_{0i} \exp(-r/a_i), \quad r_1 < r < r_2; \quad (14)$$

$$\bar{\epsilon}''_c = \frac{1.3 \cdot 10^{-25} \frac{P_0 T}{T_0} \left\{ I \sum \frac{K_{i1}}{1/\tau_D - I K_{i2}} [\exp(-r K_{i2}) - \exp(-r/I \tau_D)] - S \sigma_y \right\}}{(r - r_1) \rho}; \quad (15)$$

$$\epsilon''_c = 1.3 \cdot 10^{-25} \frac{P_0 T}{T_0 \rho} \sum \frac{K_{i1}}{1/\tau_D - I K_{i2}} \left[\frac{1}{\tau_D} \exp(-r/I \tau_D) - I K_{i2} \exp(-r K_{i2}) \right]. \quad (16)$$

II. Numerical Calculations. To determine ϵ_c and $\bar{\epsilon}_c$ one has to calculate $n = f(r)$ from (3) and find r_1 and r_2 . These calculations were performed with the σ_y of [8] for low-density polyethylene and for aloton (the commercial name for a form of polyethylene in the USA), $\rho = 0.92 \times 10^3 \text{ kg/m}^3$, together with the diffusion coefficient D and the solubility constant S taken from [9] for the range from -10 to $+60^\circ\text{C}$ without extrapolation to the range $-80 < T < +100^\circ\text{C}$. From the values of D we derived the diffusion relaxation time τ_D taking $1/\tau_D \sim D$ with normalization to the experimental value $\tau_D = 3 \times 10^3 \text{ sec}$ [5] for $T = 60^\circ\text{C}$. The values of the reaction constants K_{i1} and K_{i2} and of the gas formation rates v_{0i} found by experiment were taken from [5] for $T = 60^\circ\text{C}$. The variations in v_{01} and v_{02} with temperature were calculated from the Arrhenius relation

$$v_{01,02} = v_{c1}(0) \exp(-E/kT),$$

where $v_{c1}(0)$ is the overall value of v_0 for cross-linking at $T = 0$, $v_{c1}(0) = 5.4 \cdot 10^{22} \text{ l/m}^3 \text{ sec}$; E is activation energy, $E = 0.24 \times 10^{-19} \text{ J}$, and k is Boltzmann's constant, $k = 1.38 \times 10^{-23} \text{ J/K}$.

Calculations were performed for temperatures of 193, 293, 333, and 373°K . The parameter was the absorbed dose rate I , which was in the range from 20 to 3000 Gy/sec. The gas concentration passes through a maximum, whose value is substantially dependent on the irradiation temperature and dose rate. For example, for $I = 640 \text{ Gy/sec}$ the value of n_{\max} falls from 27×10^{25} to $2.6 \times 10^{25} \text{ l/m}^3$ as the temperature falls from 373 to 193°K , which is due to a fall in the gas formation rate. As the absorbed dose rate increases, there are increases in the dose corresponding to n_{\max} : from 0.5 MGy at $I = 20 \text{ Gy/sec}$ to 7 MGy at $I = 1000 \text{ Gy/sec}$. Figure 3 shows some of the results.

The scope for producing gas bubbles is determined primarily by I . At each irradiation temperature there is a threshold value I^* above which the gas pressure supports the forma-

tion of bubbles. Figure 4 shows I^* as a function of T . We have $I^* \cong 10^4$ Gy/sec for $T = 193^\circ\text{K}$. The values of r_1 corresponding to I^* increase as the temperature falls. For example, as T goes from 373 to 293°K , r_1 increases from 0.5 to 2.8 MGy. The values of r_1 for a given temperature gradually decrease for $I > I^*$. For example, r_1 at $T = 293^\circ\text{K}$ decreases from 2.8 to 1.1 MGy as I increases from 350 to 1000 Gy/sec.

Bubble formation extends to very high doses, and r_2 increases considerably with I and irradiation temperature (Table 1).

The calculated r_1 agree well with the data of [10] for polyethylene irradiated at $T = 353^\circ\text{K}$ ($I = 200$ Gy/sec), where above $r = 2$ MGy it was found that there were small pores, while at higher doses (up to 50 MGy) there were also cracks, whose number and size increased with dose. On the other hand, specimens irradiated at $T = 303^\circ\text{K}$ ($I = 13$ Gy/sec) did not have pores and cracks at any dose up to 50 MGy.

According to [5], gas bubbles are not formed in the surface layer $\delta = (2DS\sigma_y\nu)^{1/2}$; our calculations showed that the effects of the boundary layer should be felt at elevated temperatures (above 300°K) only at small I (below 100 Gy/sec). However, in that case bubbles are usually not formed because of the low gas pressures.

The effects of the boundary layer are very large at low temperatures (about 190°K): $\delta = 15$ mm at $I = 270$ Gy/sec, but then $I^* \geq 10^4$ Gy/sec. This effect can be neglected under real stationary irradiation conditions.

These values of r_1 and r_2 were used with the gas formation and diffusion parameters in (13)-(16) to calculate ϵ_c' , $\bar{\epsilon}_c'$, ϵ_c'' , and $\bar{\epsilon}_c''$; Table 2 gives some values of ϵ_c' . For $T = 333^\circ\text{K}$ and $I = 100$ Gy/sec, the value of ϵ_c' is 0.24% in the range 0.5-4 MGy.

The lower bound $\bar{\epsilon}_c''$ was estimated only for $r = r_1$, where it takes the maximum value. Then ϵ_c'' falls considerably and changes in sign in the range r_1 - r_2 . For $T = 333^\circ\text{K}$, ϵ_c'' varies from 0.043% for $I = 100$ Gy/sec ($r_1 = 0.5$ MGy) to 0.24% at $I = 1000$ Gy/sec ($r_1 = 0.2$ MGy). For $T = 373^\circ\text{K}$, ϵ_c'' was 0.54% for $I = 1000$ Gy/sec ($r_1 = 0.05$ MGy).

This means that the reversible changes in polyethylene are extremely small and do not exceed 1% in relative magnitude under static irradiation conditions. The changes increase with temperature and absorbed dose rate. The temperature dependence of ϵ_c' and ϵ_c'' is determined primarily by the increase in gas formation rate and the reduction in the threshold absorbed dose r_1 . The latter factor is responsible for the dose-rate dependence to a considerable extent.

Substantial values of ϵ_c' and ϵ_c'' are to be expected on pulsed irradiation of polymers at elevated temperatures.

We also calculated the relative specific-heat changes from data on the microstructure by means of (12). The amount of input heat in gas bubble formation was determined as

$$Q = 3.5 \sigma_y \rho_b = 2.63 \sigma_y \rho_s^{3/2}.$$

From the data of [7] we calculated ϵ_c from

$$\epsilon_c = \frac{\partial Q / \partial \tau}{\partial R / \partial \tau} = \frac{3.95 \sigma_y \rho_s^{1/2} d\rho_s / d\tau}{I\rho}.$$

The maximum value of ϵ_c at $r = 0.02$ MGy was 3.3%. However, firstly the authors of [7] were not convinced as to a radiolytic origin for the pores and cracks. Secondly, in accordance with Fig. 3a the gas pressure is much less than P_{CR} at 293°K and a dose rate $I = 2$ Gy/sec. This makes the result obtained for ϵ_c very doubtful.

The data of [5] have been used in estimating $Q/R = 0.19\%$ for $r = 10$ MGy and $T = 333^\circ\text{K}$, which agrees with the analytical calculation.

In all the above calculations, it will be necessary to incorporate the effects of irradiation on parameters such as the yield point, solubility, diffusion coefficient, and yields of radiolysis products. As the values for the reversible effects are extremely small, this correction was not applied, since it would not have a substantial effect on the final result.

The results are of some interest from the viewpoint of estimating the stored radiation energy in polymers. We have given experimental data on this [10], in particular for poly-

ethylene. We are concerned with the proportion of the absorbed energy that is not converted to heat and that is consumed for example in structural transformations. For most cases, this proportion $\overline{\Delta E}$ is estimated for polyethylene as 2-3%. In [11] we obtained an upper limit $\Delta E = 0.7 \pm 0.3\%$.

In polyethylene, a substantial proportion of this energy evidently accumulates in the radiolytic gases. Consequently, the reversible specific-heat changes constitute an appreciable part of the stored energy or virtually all of it from the viewpoint of the energy balance. From this viewpoint, the value of $\overline{\Delta E}$ found in [11] agrees well with calculations on ϵ_c . The magnitude of the reversible effects should increase substantially for high-density radiolytic gases.

We have developed a method of measuring the specific heat during irradiation. See [12] for a description of this method and measurements on the reversible radiation effects in the specific heat for various polymers.

NOTATION

I, absorbed dose rate, Gy/sec; I*, threshold absorbed dose rate, Gy/sec; r, absorbed dose, Gy; T, temperature, °K; σ_y , yield point, Pa; S, gas solubility, l/m³·Pa; P_{CR}, critical pressure, Pa; n, gas concentration, l/m³; N, amount of released gas, l/m³; v_i , gas formation rate in reaction i, l/m³; τ , time, sec; τ_v , gas release relaxation constant, sec; τ_D , diffusion relaxation constant, sec; a , gas formation relaxation absorbed dose, Gy; v, volume of gas bubbles in relative units; K_{i1}, K_{i2}, constants of gas formation and recombination i, l/Gy·m³ and l/Gy respectively; Q and q, amounts of heat supplied, J/m³; P₀, T₀, pressure and temperature under normal conditions; ρ , polymer density, kg/m³; ρ_s , ρ_v , surface and volume densities of pores; ϵ , ϵ_c , instantaneous and mean-integral change in specific heat; ΔE , relative accumulated energy.

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