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Optical and scintillation properties of stilbene polycrystalline and composite materials

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The new types of organic radioluminescent materials, namely, polycrystalline and composite scintillators are studied. The results of measurements of light transmittance in the range of their luminescence and transparency are presented. The original data of light-collection coefficients, the absolute light yield, the total number of scintillation photons and the radioluminescence energy yield for organic heterogeneous scintillators irradiated by ionizing radiations of different types and energies are obtained. The comparison between the characteristics of the new types of radioluminescent materials and the structurally perfected single crystals are the base of the analysis. The scintillators on the base of stilbene are the object of investigation.

Keywords: radioluminescence, optical transmittance, polycrystal, composite scintillator, light yield.

Исследованы новые типы органических радиолюминесцентных материалов — поликристаллические и композиционные сцинтилляторы. Представлены результаты измерений их оптического пропускания в области люминесценции и прозрачности. Впервые проведен расчет коэффициентов светособирания в этих гетерогенных органических сцинтилляторах, их абсолютного светового выхода, числа фотонов в импульсе радиолюминесценции и энергетического выхода радиолюминесценции для ионизирующих излучений разных типов и энергий. Анализ полученных данных основывается на сравнении свойств новых типов радиолюминесцентных материалов и структурно-совершенных монокристаллов. Исследования проведены для сцинтилляторов на основе стильбена.

Оптичні та сцинтиляційні властивості полікристалічних і композиційних матеріалів на основі стильбену. *М.З.Галунов, О.А.Тарасенко, В.О.Тарасов*.

Досліджено нові типи органічних радіолюмінесцентних матеріалів — полікристалічні і композиційні сцинтилятори. Представлено результати вимірювань їх оптичного пропускання в області люмінесценції та прозорості. Вперше проведено розрахунок коефіцієнтів світлозбирання в цих гетерогенних органічних сцинтиляторах, їх абсолютного світлового виходу, числа фотонів в імпульсі радіолюмінесценції та енергетичного виходу радіолюмінесценції для іонізуючих випромінювань різних типів і енергій. Аналіз отриманих даних грунтується на порівнянні властивостей нових типів радіолюмінесцентних матеріалів і структурно-досконалих монокристалів. Дослідження проведено для сцинтиляторів на основі стильбену.

1. Introduction

The ability of organic molecular materials to luminance under an ionizing radiation is widely used in problems of radiation equipment, radiobiology, radioecology, and radiation medicine. In recent years, the new types of organic luminescent materials, namely, organic polycrystalline and composite scintillators have been developed. In contrast to classical organic single crystals, the proposed technology allows obtaining heterogeneous objects of arbitrary shape and

unlimited size. Crystalline granules are used as a luminescent material in these types of detectors [1–4].

The total number of photons in a radioluminescence pulse determines a luminescent response of a scintillation material excited by an ionizing particle. This response is very different for different types of ionizing radiations [1]. The total number of photons of a radioluminescence pulse that incident on an optical photodetector will be determined by the optical properties of a scintillation material, that are the value of transmittance coefficient and some aspects of light passing through a medium. The optical and scintillation properties of organic single crystals are well known [1], whereas for the new types of organic heterogeneous scintillators these data are limited or are not available.

In this paper, the original data of light-collection coefficients for organic heterogeneous scintillation materials are proposed. These results are used as the base for calculation their light yield and the radioluminescence energy yield for different types of excitation. The obtained data are compared with the results of the similar studies of organic single crystals of the same chemical composition [5, 6].

2. Experimental

2.1. Objects of investigation

Crystalline granules of stilbene were obtained by grinding under liquid nitrogen structurally perfect stilbene single crystals, which had been grown from the melt. The fractions of granules with required sizes were selected using the set of calibrated sieves. To obtain polycrystals the granules with linear dimension L in the range from 2.0 to 2.5 mm were used, and for composite scintillators the granules with L from 1.7 to 2.2 mm were used. These ranges of L correspond to the optimal scintillation characteristics of the samples of polycrystals and composite detectors [7].

The samples of polycrystals were prepared by the hot pressing of granules at the pressure of 30 MPa and the temperature of 100°C. The samples were kept under pressure during 1 h, after that the pressure was gradually reduced to zero during 2 h. The samples of stilbene polycrystals with the diameter d = 30 mm and the heights h = 3, 5, 7 and 10 mm were obtained. To obtain composite scintillators the crystalline granules were introduced into a two-component gel composition Sylgard-527 [7]. The composite scintillator was placed in a housing, which was made of light guide material (acrylic plastic). We studied the samples of composite scintillators of a cylindrical shape with the inner diameter d = 25.6 mm and the inner heights h = 2.5, 5, 10 and 20 mm. In contrast to polycrystalline, where it was possible to change the size of the granules due to their sintering, in composite scintillators the initial size of granules was not changed.

As the reference scintillator we used the structurally perfect single crystal of stilbene (grown from the melt) with d = 30 mm and h = 5 mm. It scintillation characteristics are recently discussed in [5, 6]. During the measurements of light output all samples of scintillators were covered (except the exit window) with a diffusive reflecting material, e.g. Tetratek (polytetrafluoroethylene) [7].

2.2. Experimental methods

To obtain a wide range of the specific energy loss (dE/dx) of an ionizing radiation we used medium energy photons of gamma radiation from ^{22}Na, ^{60}Co, ^{137}Cs, and ^{152}Eu radionuclide sources ($E_\gamma \sim 10^5$–10^6 eV), conversion electrons from a ^{137}Cs source (E_e = 0.622 MeV), alpha particles in the energy range from 0.4 to 7.5 MeV, fast neutrons from a ^{239}Pu–Be source [5, 6]. Alpha particles in this energy range were obtained by using the attenuation of alpha particles from ^{239}Pu (5.15 MeV) and ^{226}Ra (7.68 MeV) sources in a layer of air. The actual energies of the alpha particles were estimated from their residual range [1, 8].

A multi-channel amplitude analyzer AMA-03F was used to measure amplitude scintillation spectra of samples. The light output of samples was calculated by comparison with the value of the light output of the reference stilbene single crystal [9, 10] whose light yield was equal to 14,700 photons per 1 MeV of gamma radiation energy. Next, to calculate the total number of photons in a radioluminescence pulse the correction for the light-collection coefficient τ, which takes into account geometry of a sample and a type of excitation, was performed [9].

To separate the spectrum of recoil protons generated by fast neutrons of a ^{239}Pu–Be radionuclide source, we used the method of discrimination of an ionizing radiation by its scintillation pulse shape and the corresponding set-up that provided this method [1]. The method of reconstruction of the

neutron spectrum from the measured recoil proton spectrum was described in detail in a number of papers (see the review in [1]).

Measurements of light transmittance T were performed by Shimadzu-2450 spectrophotometer with the integrating sphere.

2.3. Aspects of light-collection modeling

The light-collection coefficient τ is defined as the ratio between the value of the light output (the total number of photons of a scintillation pulse that incident on a photodetector from a scintillator) and the value of the light yield (the total number of photons produced in the scintillator) [1, 9]. The light-collection coefficient τ was calculated by the simulation of light propagation through a scintillator.

The simulation of light propagation in heterogeneous scintillator was performed by Monte Carlo method. The general approach used during the simulation is described in [10]. In the case of heterogeneous systems, a light scattering in the bulk of a scintillator was additionally simulated. Currently, two approaches are commonly used for modeling in such systems: 1) light propagation through the system with a large number of optical interfaces with reflection and refraction on them by Fresnel's law is considered [11], 2) a solid scattering medium is considered by applying alternating variations of a photon path length before scattering and a scattering direction for the specified phase scattering function [12].

In this paper, we used the second approach. Variations of path length before scattering was performed using the coefficients of optical absorption k_a and scattering k_s. These coefficients were found from the measurement of transmittance T in the different regions of the spectrum for the samples with varying heights. It was assumed that a change in transmission is only due to light scattering for $\lambda = 700$ nm and a change in transmission is defined both light scattering and light absorption for the wavelength which corresponds to a maximum in the luminescence spectrum of a scintillator ($\lambda = 390$ nm for stilbene [7, 13]). Accordingly, the coefficient k_a was calculated as the difference between the total coefficient for $\lambda = 390$ nm and the coefficient for $\lambda = 700$ nm.

Using the values of the coefficients k_a and k_s the total probability of "elimination" of a photon from the flux in the result both scattering and absorption was initially determined, whereat the point of interaction was determined too. Then the type of interaction, i.e. absorption or scattering, was varied. A "death" of a photon was recorded in the case of absorption, whereat a "fate" of the next photon was tracked. The scattering angle was varied using the phase scattering function. In this paper, we used the Henyey-Greenstein scattering function [14, 15]:

$$p(\cos j) = \frac{1}{4\pi} \cdot \frac{1 - g^2}{(1 + g^2 - 2g\cos j)^{3/2}}. \quad (1)$$

In (1) the g-value completely determines the scattering properties of a material and ranges from -1 to 1, j is the angle between the incident and the scattered light beams, and $p(\cos j)$ is the probability of scattering by the angle j. If $g = -1$, then the scattering will occur predominantly "back". If $g = 0$, the scattering will occur uniformly in all directions. If $g = 1$, then the scattering will occur "forward". This function allows by selection the g-value (which may be a random number distributed in a certain interval) to obtain preferential scattering directions.

In this paper, the selection of the parameter g was carried out in such a way that the dependence between the transmittance T and height h of the samples (which is obtained by simulation of the propagation of the external light flux) corresponds to the experimental dependence. The values of g were used to determine the coefficient τ when the scintillations "born" in various points of the sample volume. This situation corresponds to the case of irradiation of the samples by the ionizing radiations with different values of dE/dx [1].

3. Results and discussion

Let us use the following notations: C is the light yield, $E_{ph}(\lambda^{av}_{em})$ is the average energy of a luminescence photon, τ_i is the light-collection coefficient, E_i is the excitation energy, P_i is the total number of photons in a scintillation pulse, and Y_i is the energy yield. The suffix i means the excitation of i type. Below by ph, γ, e, n, and α we denote the following types of excitation i: light photons, photons of gamma radiation, conversion electrons, neutrons and alpha particles, respectively. The symbol of averaging will be omitted.

The average value of the radioluminescence energy yield for a particle with the initial energy E_0 crossing the maximum

range in a scintillator is defined as [5, 6]:

$$\langle Y_r \rangle = \frac{1}{E_0} \int_0^{E_0} Y_r(E)dE = \frac{L}{E_0}, \quad (2)$$

where L is the total energy of radioluminescence photons. The L-value for the excitation of i type will be calculated as $P_i \times E_{ph}(\lambda^{av}_{em})$, where the value of $E_{ph}(\lambda^{av}_{em})$ for the single crystal of stilbene was obtained in [13] from its photofluorescence spectrum.

3.1. Light yield and light-collection coefficients

Fig. 1 shows the results of measurements of transmittance T for polycrystalline and composite stilbene scintillators of different height h. The measurements were carried out at the wavelengths those correspond, for stilbene, to the maximum values of absorption (360 nm), luminescence (390 nm), as well as the spectral band of transparency (700 nm). We used the T-values to determine the coefficients of optical absorption k_a and scattering k_s according to the above-described procedure.

The calculated values of k_a and k_s for different heterogeneous scintillators are presented in Table 1. The coefficients k_a and k_s for the single crystal were earlier obtained [6, 10] when calculating the light-collection coefficients.

The obtained values of k_a and k_s were used to calculate the light-collection coefficients τ_γ, τ_e and τ_α for polycrystalline and composite scintillators of different height h. For these calculations we took into account the fact that the range of an alpha particle with the energy E_α of 5 MeV is approximately equal to 30 μm, and for an electron with the energy E_e of 0.622 MeV is approximately 2 mm. Values of ranges for alpha particles and electrons were calculated using the online programs ASTAR and ESTAR of the National Institute of Standards and Technology (NIST) [16]. Thus,

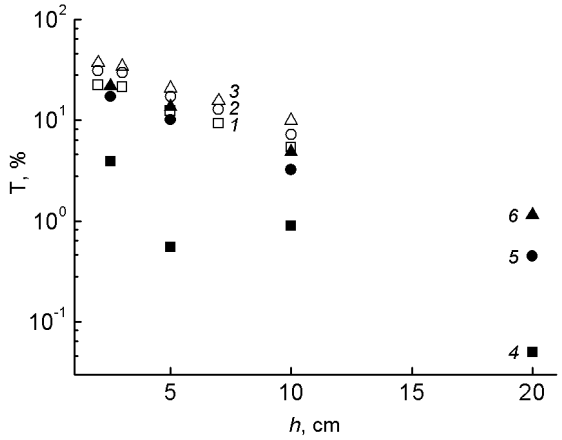

Fig. 1. Transmittance T of stilbene polycrystalline (*1*, *2* and *3*) and composite (*4*, *5* and *6*) scintillators of height h. The measurements were carried out at wavelength λ: *1* and *4* for λ = 360 nm, *2* and *5* for λ = 390 nm, *3* and *6* for λ = 700 nm.

these radiations lose their energy within the single granule. The location of the scintillation pulse was considered equally probable over the sample volume for the cases of gamma- and neutron excitations. In this situation, the radioluminescence occurred in different granules of a heterogeneous sample. This assumption is valid, since the penetration depths of gamma radiation photons and fast neutrons for the experimental energy ranges of E_γ and E_n may be even greater than the height h of the experimental samples [1, 8]. Therefore, the value τ_n was taken equal to τ_γ.

The calculated values of the light-collection coefficients τ_γ, τ_e and τ_α for organic scintillators on the base on stilbene are presented in Table 2. The values for the single crystal are taken from [10]. The study of composite scintillators for the case of the excitation by conversion electrons did not carried out because the energy resolution of the peaks of conversion electrons for these objects was very large. This reduced an accuracy of the determination of the scintillation pulse amplitudes.

Table 1. Coefficients k_a and k_s for single crystalline, polycrystalline and composite scintillators on the base of stilbene

Scintillator	$k_a + k_s$, cm^{-1} (390 nm)	k_s, cm^{-1} (700 nm)	k_a, cm^{-1}
Single crystal	0.048	0.039	0.009
Polycrystal	1.879	1.709	0.170
Composite scintillator	2.006	1.617	0.389

Table 2. Values of C, τ_γ, τ_e and τ_α for heterogeneous scintillators on the base of stilbene

Scintillator	d, mm	h, mm	C, photons/MeV	τ_γ	τ_e	τ_α
Single crystal	30	5	14.700	0.659	0.649	0.635
Polycrystal	30	3	14.801	0.735	0.838	0.831
-//-	30	5	15.436	0.669	0.728	0.718
-//-	30	7	15.960	0.610	0.604	0.594
-//-	30	10	15.105	0.540	0.413	0.415
Composite scintillator	25.6	5	13.524	0.515	–	0.459
-//-	25.6	10	14,505	0.372	–	0.310
-//-	25.6	20	13,629	0.231	–	0.158

The light yield C of the samples was calculated by dividing the light output by the coefficient τ_γ. The light output, in turn, was determined by the comparison method [9, 10] with the value of the light output of the reference single crystal of stilbene. We used for comparison the position of Compton edges in the amplitude scintillation spectra of samples excited by photons of gamma radiation of ^{137}Cs. The C-values are also shown in Table 2.

Using the results of measurements of the amplitude scintillation spectra and the values of C, τ_γ, τ_e and τ_α we calculated the total number of photons P_i in the scintillation pulse and the radioluminescence energy yield Y_i for organic heterogeneous scintillators excited by the ionizing radiations with low and high values of dE/dx. The calculation procedure was described previously in [5, 6].

3.2. Low dE/dx

Fig. 2 shows the calculated values of the number of scintillation photons P_γ and P_e for the cases of the excitation of organic scintillation materials by photons of gamma radiation and conversion electrons, respectively. Secondary conversion electrons initiated the scintillations in the case of gamma photon excitation. Within the discussed energy range the plot of P_γ as a function of E_γ yields a straight line. For conversion electrons the calculation was performed only for a specific value, which corresponded to the energy of the incident electron E_e. The energy $E_e = 0.613$ MeV corresponds to the experimental conditions. To calculate this value we take into account that before the electron with the initial energy $E_e = 0.622$ MeV strikes a sample it has to pass through a layer of air about 4 cm thick.

The Y_γ-value, according to (2), is equal to the product of a slope of the curve (see. Fig. 2) by $E_{ph}(\lambda^{av}{}_{em})$. The value Y_e is obtained as

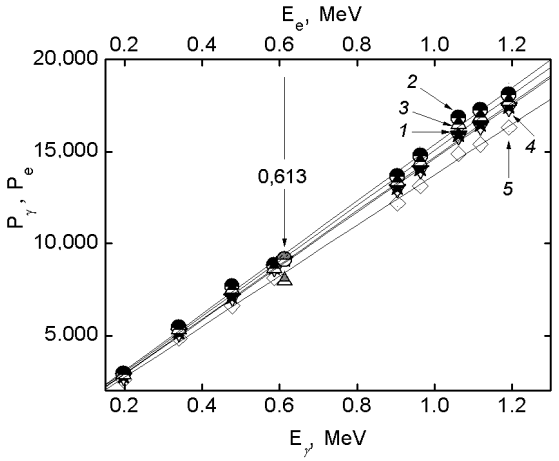

Fig. 2. The total number of photons P_γ and P_e in a scintillation pulse for scintillators on the base of stilbene excited by photons of gamma radiation and conversion electrons, respectively. Points are the experimental values; the solid lines are the calculated linear dependences of P_γ against E_γ. The arrow indicates the energy $E_e = 0.613$ MeV of the conversion electrons. The following notations are used: *1* is the single crystal; *2* and *3* are polycrystals with $h = 5$ and 10 mm, respectively; *4* and *5* are composite scintillators with $h = 10$ and 20 mm, respectively.

$P_e \times E_{ph}(\lambda^{av}{}_{em})/E_e$ for $E_e = 0.613$ MeV. The results of calculations of Y_γ- and Y_e-values for heterogeneous detectors on the base on stilbene are presented in Table 3.

From the results shown in Fig. 2 and Table 3, we obtain the following. There is a very slight difference in the values of P_γ and P_e (and, consequently, according to (2), in the values of Y_γ and Y_e) for all studied types of scintillation materials on the base on stilbene. Taking the value of P_i for the reference single crystal equal to 100 %, we will characterize a spread of P_i- (Y_i-) values in the form:

Table 3. Values of Y_γ and Y_e for heterogeneous scintillators on the base of stilbene

Scintillator	d, mm	h, mm	Y_γ	Y_e
Single crystal	30	5	0.0453	0.0456
Polycrystal	30	3	0.0456	0.0543
-//-	30	5	0.0475	0.0458
-//-	30	7	0.0492	0.0428
-//-	30	10	0.0465	0.0400
Composite scintillator	25.6	5	0.0416	–
-//-	25.6	10	0.0447	–
-//-	25.6	20	0.0420	–

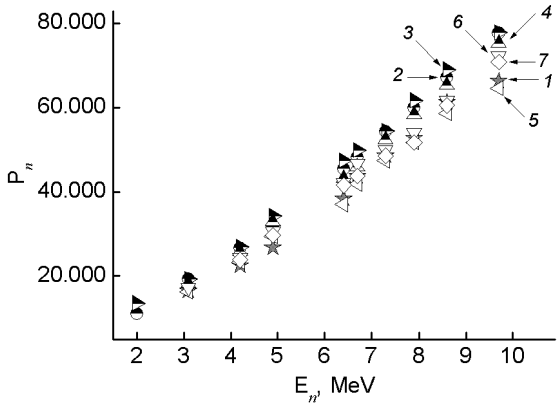

Fig. 3. The total number of photons P_n in a scintillation pulse for scintillators on the base of stilbene excited by fast neutrons with the energy E_n. The following notations are used: *1* is the single crystal; *2*, *3* and *4* are polycrystals with h = 5, 7 and 10 mm, respectively; *5*, *6* and *7* are the composite scintillators with h = 5, 10 and 20 mm, respectively.

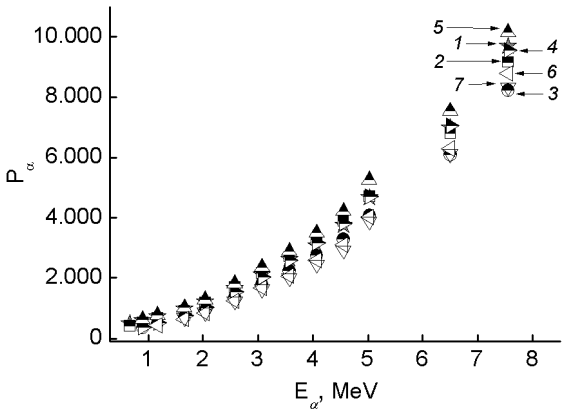

Fig. 4. The total number of photons P_α in a scintillation pulse for scintillators on the base of stilbene excited by fast neutrons with the energy E_α. The following notations are used: *1* is the single crystal; *2*, *3*, *4* and *5* are polycrystals with h = 3, 5, 7 and 10 mm, respectively; *6* and *7* are composite scintillators with h = 5 and 10 mm, respectively.

$$\Delta_i = \frac{|P_i^{samp} - P_i^{ref}|}{P_i^{ref}} \cdot 100\%, \qquad (3)$$

where P_i^{samp} and P_i^{ref} are the values of P_i for the sample of a heterogeneous scintillator and for the reference single crystal, respectively.

According to the above-mentioned results (see Fig. 2 and Table 3), the absolute values of P_γ and P_e (Y_γ and Y_e) for organic polycrystalline and composite materials are both slightly higher and slightly lower than the corresponding values for the reference single crystal. The values of Δ_γ and Δ_e did not exceed, respectively, 9 % and 20 % for all the samples under investigation.

3.3. High dE/dx

Fig. 3 shows the total number of photons P_n in the scintillation pulse for the case of excitation of organic scintillators on the base on stilbene by fast neutrons. In this case, recoil protons excite the scintillations. The P_n-values were calculated for the energies E_n of the neutron spectrum of ^{239}Pu–Be, obtained by the reconstruction procedure of the corresponding experimental spectrum of the recoil protons [1].

The calculated values P_α for alpha particles in the energy range E_α from 0.4 to 7.5 MeV are presented in Fig. 4. Expectedly, for the ionizing radiations with high values of dE/dx such dependence is non-linear. In this case, the non-linearity in both cases increases with dE/dx, i.e. from fast neutrons to alpha particles.

Additionally, the data presented in Figs. 3 and 4 do not find any features in the P_n- and P_α- values for the new types of organic scintillators in comparison with the

single crystal. As for the ionizing radiations with low dE/dx, the P_n- and P_γ-values weakly and randomly changes relatively to the corresponding values for the reference material. Within the experimental series of organic heterogeneous scintillators (see. Table 2) the maximum value of Δ_n (3) is about 18 % for E_n = 9.7 MeV and the maximum value of Δ_α (3) is about 15 % for E_α = 7.56 MeV. A slight increase of the Δ_n- and Δ_α-values was observed for the initial regions of the experimental values of E_n and E_α. Most likely, this is caused by increase in the error of identification of positions of maxima in amplitude scintillation spectra for a small total number of photons in a scintillation pulse.

3.4. Radioluminescence energy yield for different types of ionizing radiations

For fast neutrons and alpha particles we calculated the values $Y_n = P_n \times E_{ph}(\lambda^{av}{}_{em})/E_n$ and $Y_\alpha = P_\alpha \times E_{ph}(\lambda^{av}{}_{em})/E_\alpha$ respectively, according to Eq.(2). The Y_n-values for the investigated range of energies E_n and samples of organic heterogeneous scintillation materials are in the range from 0.016 to 0.025, while in the case of alpha excitation the calculation gives the values of Y_α from 0.0012 to 0.0040.

Fig. 5 summarizes the dependence of the radioluminescence energy yield of organic scintillation materials as a function of dE/dx. To demonstrate these results we randomly selected one sample from each of the studied types of scintillators: single crystal, polycrystalline and composite scintillators. Values of dE/dx were calculated using the online programs ESTAR, PSTAR, and ASTAR of NIST [16]. The calculated values of Y_γ (see Table 3) are also presented for comparison. In the case of neutron excitation we calculated the values of dE/dx for the maximum energy of recoil protons, which were generated by the corresponding fast neutrons with a set of energies E_n.

The results of the investigation of the radioluminescence energy yield for different types of organic scintillation materials confirm the common regularity of the energy losses in tracks of ionizing particles with different values of dE/dx. Previously [5, 6], we have identified such a regularity for organic single crystalline scintillators. The radioluminescence energy yield reduces by a factor of 2–3 in going from the ionizing radiations with low dE/dx (values Y_γ and Y_e) to fast neutrons (values Y_n). Further increase

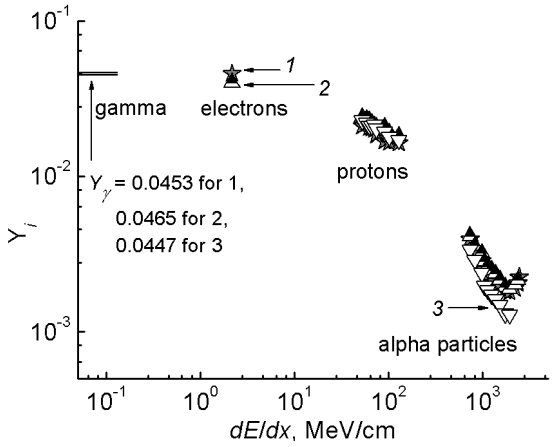

Fig. 5. The radioluminescence energy yield Y_i of scintillators on the base of stilbene as a function of dE/dx of the ionizing radiation.

The following notations are used: *1* is the single crystal, *2* is the polycrystal with h = 5 mm, *3* is the composite scintillator with h = 5 mm. Solid lines indicate the position of the Y_γ-values in the scale of Y_i for samples *1*, *2* and *3*.

in dE/dx leads to a more precipitate drop in the radioluminescence energy yield. For the case of alpha excitation, the Y_α-values are 10–40 times less than the $Y_{\gamma-\text{ and}}$ Y_e-values. Using the scintillation materials on the base on stilbene as an example (see. Fig. 5), it is obviously that this tendency holds true both for structurally perfect single crystals and for the new types of organic heterogeneous scintillators.

The complicated dependence of Y_α versus dE/dx (see. Fig. 5) in the range of large values of dE/dx (or small values of E_α) has been discussed previously [5, 6]. The effect of an ion recharge can be the reason of this dependence [1, 17]. According to [17], this effect takes place for alpha particles with the energies about 1 MeV.

In our opinion, slightly lower values of Y_α (P_α) obtained for composite samples in comparison with the single crystal and polycrystalline samples (see. Fig. 5) may be explained by the technology of production of this type of material. A thin layer of transparent gel composition, which is used for binding of granules [7], may be formed on the surface of such a scintillator during it production. In this case, the actual energy of the alpha particles passed through this layer will be less than the initial one. This effect will increase with decreasing the initial energy E_α.

4. Conclusions

The original data of the light-collection coefficients for the new types of organic materials, i.e. polycrystalline and composite were obtained. This allowed calculating the light yield C and the radioluminescence energy yield Y_i for these types of detectors.

The analysis of the experimental results did not find any significant differences in the values of C, P_i and Y_i for organic single crystal and for heterogeneous scintillation materials excited by ionizing radiations of different types and energies. This allows us to extend the mechanisms of the energy losses, which we recently proposed [6], to a wider class of organic scintillation materials.

Inconsiderable differences in the values of C, P_i and Y_i do not depend on the aspects of energy exchange processes in different types of organic scintillators. These differences may be caused by technological and methodical features of production and study of experimental samples, respectively. They are the following:

— the methodical error in determining the relative light output of an experimental sample, especially when scintillation pulses of low amplitudes are detected;

— a number of assumptions in calculating the light-collection coefficients by the methods of mathematical modeling;

— the spread of the technological parameters used for production the scintillation samples of the same type (for example, according to [10], the difference in the light output of the samples of stilbene single crystals of the same dimensions and produced by the same technology, is about 10 %);

— some aspects of obtaining an organic heterogeneous scintillation material.

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