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HETEROSTRUCTURED SCINTILLATORS BASED ON ORGANIC SINGLE CRYSTAL GRAINS FOR A HIGH PULSE SHAPE DISCRIMINATION

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This article is related to studying the properties and improving the characteristics of the new types of organic heterostructured scintillators as detectors of fast neutrons and short-range charged particles. This research is aimed to find the opportunities to improve the characteristics of the detector, based on a heterostructured scintillation material, and to expand the possibilities of its practical applications, in particular, for the tasks of separate detection of ionizing radiations of different types.

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INTRODUCTION

A detection of low-intensity ionizing radiation fluxes is very relevant. It is important in the modern environmental, biological, medical, and geological problems. It is used to prevent the unauthorized transportation of radioactive materials and to eliminate the consequences of human-made disasters. The product of the dose value of radiation absorbed by the body on the value of the radiation weight factor ω_R is the quantitative characteristic that determines an influence of an ionizing radiation on the human body. The ω_R-value determines the long-term effects (the possibility of serious diseases such as oncology) of a chronic dose of low intensity.

The main part of the neutron energy spectrum of natural radionuclides or possible radiations around nuclear power plants, which occur due to failure, has the energy $E_n \leq 2$ MeV. If for photons of gamma radiation the value of ω_R is equal to 1, then for alpha particles, fast neutrons with energies $E_n \leq 2$ MeV, and nuclei of heavy ionized atoms, the value of $\omega_R = 20$ [1]. It means that if the device accurately counts the number of registered events without dividing them by their nature, then such information does not correspond to the real situation, and its use can be dangerous.

The precise neutron spectrometry requires using the scintillation materials with high hydrogen proportion. Therefore, organic molecular materials become optimal. Due to *A* and *Z* small values for carbon and hydrogen nuclei, these materials with a density close to 1 g/cm^3 have negligible backscattering for charged particles. It increases an efficiency and stability of the detection of charged particles in comparison with inorganic scintillators. Unfortunately, the technology of obtaining organic single crystals imposes strict limits on their maximum size and shape, besides it is quite expensive. Liquid scintillators are toxic and flammable [2].

To solve this problem, we have proposed to study more promising newly created materials. It is a heterostructured scintillation material of new types [2–6]. They are organic scintillators containing single crystal grains. When we are sintering the grains by hot pressing, we obtain a polycrystal (Van-der-Waltz ceramics). The introduction of the grains into a transparent gel composition gives a composite scintillator. Ionizing radiations generate two types of luminescent response. They are the prompt and the delayed radioluminescence. The ionizing radiation with high values of specific energy losses (*dE/dx*) (recoil nuclei, alpha particles, and low-energy electrons) generates a scintillation pulse with a large contribution of a slow component. The radiations with a small *dE/dx* (e.g., photons, X-rays, photons of gamma radiation, electrons of medium and high energies) generate a scintillation pulse whose slow component contribution is small. The slow component arises as a result of the process of triplet-triplet (*T*-*T*) annihilation of localized triplet molecular excitons (in the future *T*-states) [2, 7, 8]. In the presence of an efficient diffusion transport of *T*-states, they can be localized on neighbouring molecules, i.e., to approach each other at a distance where the exchange resonance mechanism of their interaction becomes possible. The efficiency of the transport of *T*-states in a scintillation material and its ability to separate signals from radiation with different *dE/dx* determines the slow component intensity. For traditional organic single crystals and liquids, their ability to separate the signals from the radiations with different *dE/dx* is a well-known experimental fact. It is the pulse shape discrimination (PSD) capability. The features of transport and recombination of *T*-states in such materials are also well studied. Such information for heterostructured materials, where a certain grain size can limit the migration length of *T*-states, is practically absent.

1. EXPERIMENTAL

1.1. EXPERIMENTAL SAMPLES

We have chosen *trans*-stilbene and *p*-terphenyl as the scintillation materials. The reference single crystal

samples were grown using the Bridgman-Stockbarger method. More details one can see in [8].

To obtain heterostructured samples based on *p*-terphenyl we used commercially available raw material, which were thoroughly purified. Unfortunately, trans-stilbene manufactured by leading companies does not allow obtaining scintillators with the required high characteristics [9]. Our preliminary studies (see, for example, [8]) have found impurities that limit the characteristics of the material. Therefore, for the production of the samples based on *trans*-stilbene we synthesized this substance of a sufficient quality.

Composite scintillators were made from crystalline grains, which were previously obtained by grinding organic single crystals under liquid nitrogen or a polycrystalline ingot, which was formed in the process of refining raw materials by the melting zone. By using the set of calibrated sieves, the crystalline grains were separated into the fractions of different sizes. Selected grain fractions were introduced into a polysiloxane elastomer matrix [10]. The resulting mixture was introduced into the container for a polymerization of the matrix and for a formation of the scintillator shape. Dense packing of crystalline grains in a composite scintillator [3, 4] was ensured by adding 70…80% of grains from the volume of the scintillator to the matrix.

For the studies using alpha particles, the surface of a composite scintillator was not covered with a layer of polysiloxane elastomer. This was achieved by applying an additional layer of dry grains. The sample was left at the temperature of 60 °C for 24 h. After complete polymerization, the composite scintillator was removed from the mold-forming container [5]. Several series of the composite scintillators based on stilbene and *p*-terphenyl with the diameter d = 18 mm and the height h = 4 mm, as well as with d = 20 mm and h = 5 mm were manufactured for this research. We have varied the sizes of the grains in the following ranges: 0.1…0.3 mm, 0.3…0.5, 0.5…1.0, 1.0…1.2, 1.0…1.5, 1.2…1.4, 1.4…1.8, 1.5…2.0, and 1.8…2.0 mm.

In contrast to single crystals and plastics, composite scintillators are not continuous media. They are closer to soft matter. As can be seen from the description of the technology of their production, they have no technological limitations on the area of the entrance window. They are cheaper than single crystals because they do not require the most expensive stage of obtaining a single crystal, namely its growth and mechanical processing (the main source of loss of single crystal material).

Polycrystalline stilbene and *p*-terphenyl scintillators were produced in a mold with an inner diameter of 15 mm. A sample of the starting material weighing 0.8 g was placed between polished metal spacers. The mold inserted into the heater was placed under a hydraulic press with a maximum force of 10 t. The heating temperature was regulated by a laboratory autotransformer and monitored by a digital thermometer with cold junction compensation and an NR-39 thermocouple. The temperature was brought to the pressing temperature by gradual heating for one hour. Polycrystals of stilbene were pressed at the temperature of 100 °C, and *p*-terphenyl at the temperature of 165 °C for one hour. The pressing pressure was 50 MPa and was applied when the temperature reached the pressing temperature. After one hour, the press rod was raised, releasing the pressure on the mold, and the heating was turned off. The polycrystal was extracted the next day [7, 8]. The following grain fractions were used to obtain samples: 0.1…0.3, 0.3…0.5, 0.5…1.0, 1.0…1.2, 1.2…1.4, 1.4…1.8, 1.8…2.0 mm. The samples of stilbene and *p*-terphenyl polycrystals with a thickness of 4 mm and a diameter of 15 mm were obtained.

1.2. LIGHT OUTPUT MEASUREMENTS

The amount of light emitted by the scintillator is characterized by the magnitude of the luminous flux, i.e. the ratio of the number of photons arising in the scintillator to the energy lost in it by an ionizing radiation. The essence of the method is to measure the electrical signal at the output of the photodetector, which registers the glow of the scintillator. At the output of the photodetector, the electrical signals, caused by the photons of the scintillation pulse that have arisen in the scintillator, are summed. The value of this accumulated signal is proportional to the amplitude of a scintillation pulse [9, 11]. For organic scintillators, it is better to use photomultipliers as a photodetector. We used a 9208A photomultiplier manufactured by Electron Tubes Ltd., for which, according to the passport data, the dark current has a very low value of $6.8 \cdot 10^{-11}$ A at an anode sensitivity of 50 A/Lm [12]. The original photomultiplier circuit had an electrical signal generation time $\tau_{ap} = 2$ μs.

More often, the light output is determined by the spectrum of scintillation amplitudes. For this task, an ADC was used, connected to a PC, where the accumulation and processing of the received data was carried out.

The relative light output can be calculated using the following formula:

$$J = \frac{J_0}{J_{ref}} \times 100\ \%, \qquad (1)$$

where J_0 is the value of the maximum amplitude of the spectrum of the test sample; J_{ref} is the value of the maximum amplitude of the spectrum of the reference scintillator. Recalculating the data obtained in relation to the value of the light output of the reference scintillators in photons per 1 MeV, we obtain the values of the light output of the studied samples. The error of this method is less than 5% [13]. The measuring setup was calibrated using ^{22}Na, ^{60}Co, ^{137}Cs, and ^{152}Eu gamma sources. ^{239}Pu was used as a source of alpha particles.

1.3. FOM MEASUREMENTS

The method of charge-time transformation lies in the studies of the peculiarities of the recombination of triplet states in scintillators. This method is the basis for calculating an important parameter called the Figure-of-Merit (FOM). This method makes it possible to find the difference between pulses created by photons of gamma radiation and fast neutrons, using the integration of certain pulse time intervals. All data are analyzed using the Charge Comparison Method (CCM) [14–18].

The pulses generated by gamma photons and neutrons look quite similar and have a fast rise time and a slow decay time. The excitation created in an organic scintillator by gamma photons decays faster than the excitation created by fast neutrons. Therefore, by the relative intensity of the tail part of the pulse, it is possible to identify the type of the particle that causes such excitation. Thus, the ratio of the area of the tail of the signal (tail integral) and the total area of the signal (total integral) is different for neutron and gamma pulses. These values were used to analyze the ability to separate detection of ionizing radiations based on the shape of the scintillation pulse.

The dependence between the separation properties on the pulse shape for different samples of heterostructured scintillators was evaluated using the *FOM*-value [17]:

$$FOM = \frac{\Delta S}{FWHM_1 + FWHM_2}, \quad (2)$$

where ΔS is the difference between peak maxima, $FWHM_1$ and $FWHM_2$ are the full width at half maximum of the gamma and neutron peaks, respectively. The *FOM* is the measure of the quality of separation of the signals caused by fast neutrons and photons of gamma radiation. The ^{239}Pu-Be source was used in our studies. This is the source of both fast neutrons and accompanied photons of gamma radiation.

1.4. LIGHT TRANSMITTANCE MEASUREMENTS

The measurements of the light transmittance *T* in the range from 300 to 700 nm were performed using Shimadzu-2450 spectrophotometer with the integrating sphere. The comparison channel remained blank and the light flux inside it was calibrated to be the same as the light flux falling on a sample in the measuring channel. The inaccuracy of the calibration was limited by 0.5%. The value of *T* was calculated as follows:

$$T = \frac{I}{I_0} \times 100\ \%, \quad (3)$$

where I_0 is the light flux in the comparison channel; *I* is the light flux, which has passed through a sample in the measuring channel. Actually, the *T*-value (3) is a relative light transmittance, where $T = 100\%$ is the luminous transmittance of air.

1.5. MEASUREMENTS OF LUMINESCENCE AND EXCITATION SPECTRA

To obtain luminescence and absorption spectra we used spectrofluorimeter Varian Cary Eclipse. In our experiments, the range of wavelengths is 300…700 nm.

2. RESULTS AND DISCUSSION

The values of the relative light output *J* for composite and polycrystalline scintillators were calculated according to Eq. (1). The results of these calculations as the function of the average size L_{av} of grains are shown in Fig. 1 for scintillators based on stilbene and in Fig. 2 for scintillators based on *p*-terphenyl, respectively. The dependence of the value of the energy resolution *R* on the average size of the grains L_{av} is shown in Fig. 3 for scintillators based on stilbene and in Fig. 4 for scintillators based on *p*-terphenyl, respectively.

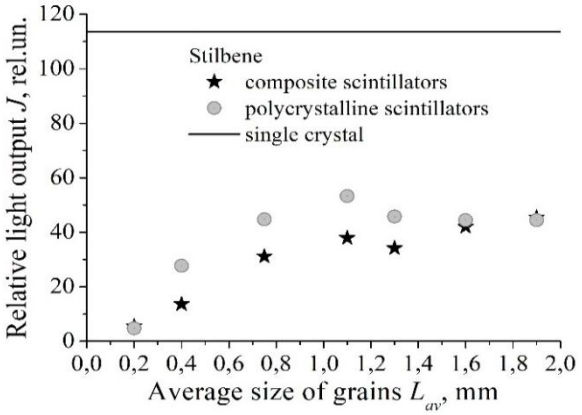

Fig. 1. Dependence of the value of the light output J on the average size L_{av} of grains for composite scintillators based on stilbene (stars), polycrystalline scintillators based on stilbene (circles) and stilbene single crystal (line)

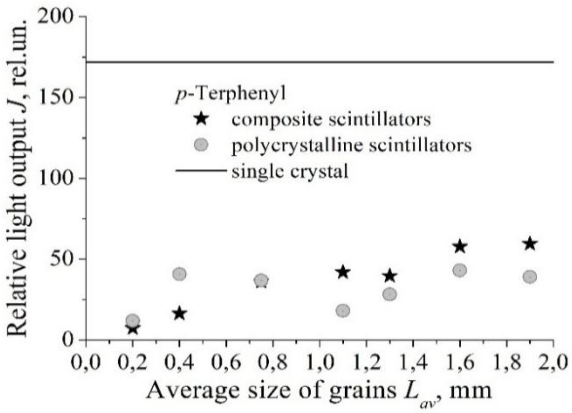

Fig. 2. Dependence of the value of the light output J on the average size L_{av} of grains for composite scintillators based on p-terphenyl (stars), polycrystalline scintillators based on p-terphenyl (circles) and p-terphenyl single crystal (line)

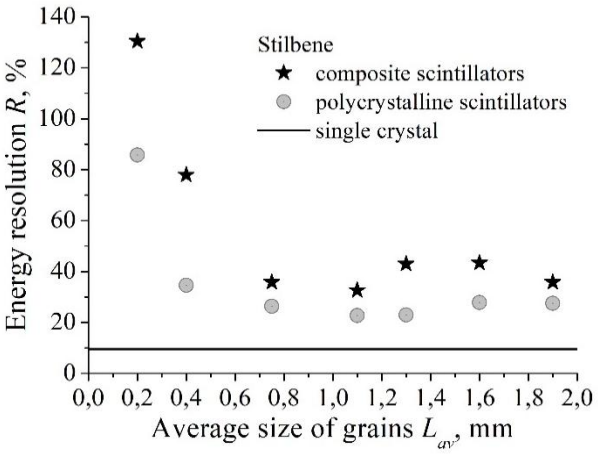

Fig. 3. Dependence of the value of the energy resolution R on the average size L_{av} of grains for composite scintillators based on stilbene (stars), polycrystalline scintillators based on stilbene (circles) and stilbene single crystal (line)

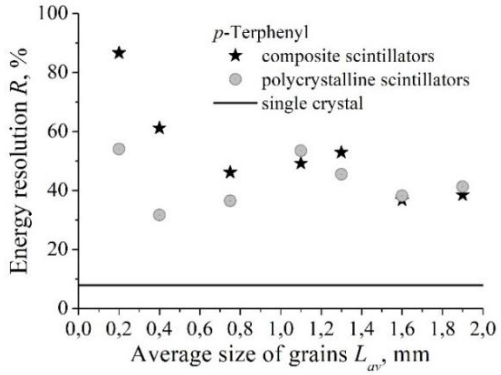

Fig. 4. Dependence of the value of the energy resolution R on the average size L_{av} of grains for composite scintillators based on p-terphenyl (stars), polycrystalline scintillators based on p-terphenyl (circles) and p-terphenyl single crystal (line)

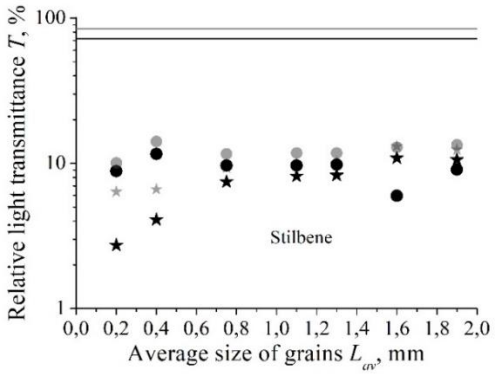

Fig. 5. Dependence of optical transmittance T on the average size L_{av} of grains for composite scintillators based on stilbene (stars), polycrystalline scintillators based on stilbene (circles) and stilbene single crystal (line). Black colour corresponds to a wavelength of 400 nm, grey to a wavelength of 700 nm

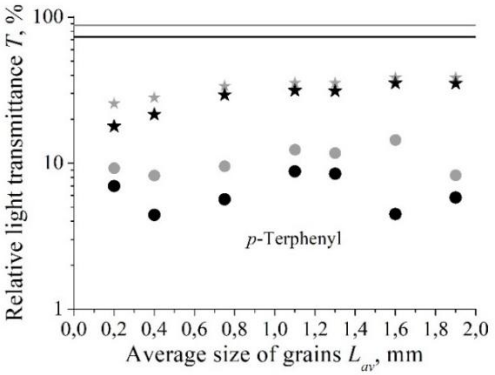

Fig. 6. Dependence of optical transmittance T on the average size L_{av} of grains for composite scintillators based on p-terphenyl (stars), polycrystalline scintillators based on p-terphenyl (circles) and p-terphenyl single crystal (line). Black colour corresponds to a wavelength of 400 nm, grey to a wavelength of 700 nm

One can see that in the case of excitation by alpha particles, composite and polycrystalline scintillators based on stilbene grains show similar dependences of the value of the relative light output *J*, as well as the energy resolution *R* on the average size of the grains. Initially, with an increase in the L_{av}-value (approximately 0.8 mm), there is a significant increase in the *J*-value and improvement in the *R*-value of the samples. For L_{av}-values greater than 0.8 mm, the light output *J* of the samples, as well as their energy resolution *R* do not change significantly.

For composite scintillators based on *p*-terphenyl, the dependences of the relative light output *J* and energy resolution *R* on the average size of grains L_{av} were observed, similar to scintillators based on stilbene grains. As the L_{av}-value increases, there is an increase in the *J*-value and an improvement in the *R*-value, which is quite noticeable for L_{av} less than 0.8 mm and becomes weak for larger grain sizes.

The situation is different for polycrystalline scintillators based on *p*-terphenyl. For these samples, we did not observed the dependence of the *J*-value and the *R*-value on the average grain size L_{av}. Such results may be related to the fact that the sintering and recrystallization processes occurring during the hot pressing process proceed differently in stilbene and *p*-terphenyl, but this issue requires further research.

For the singlecrystalline, composite, and polycrystalline scintillators based on stilbene and *p*-terphenyl under investigation, measurements of the value of the optical transmission coefficient *T* were made relative to air in the wavelength range of 190…800 nm. The dependences of the *T*-value on the average size L_{av} of grains for the wavelengths of 400 and 700 nm were determined. These dependencies are shown in Fig. 5 for scintillators based on stilbene and in Fig. 6 for scintillators based on *p*-terphenyl, respectively. These wavelengths were chosen for the following reasons. The wavelength of 400 nm lies in the luminescence range of the organic scintillation materials, so it can be used to simulate the passage of scintillation photons through a sample. The wavelength of 700 nm corresponds to the region of transparency of these materials and can be used to assess the influence of the light scattering process in a sample.

As it expected, the obtained results showed that the values of optical transmittance *T* of composite and polycrystalline scintillators are quite low compared to single crystals, which is due to the diffuse nature of light passing through them. The dependences of the *T*-value on the L_{av}-value are similar for composite scintillators based on stilbene and based on *p*-terphenyl. Both in the case of stilbene and in the case of *p*-terphenyl, with an increase in the L_{av}-value, there is a gradual increase in the *T*-value and a decrease in the difference between the values of *T* that were measured for the wavelengths of 400 and 700 nm for the same sample. For small L_{av}-values (less than 0.8 mm), such an increase in the value of *T* and a decrease in the difference between the values of *T* for the wavelengths of 400 and 700 nm are quite noticeable, and for values of L_{av} greater than 0.8 mm, these changes become insignificant. Such results are in a good agreement with the dependences of *J*-value on the L_{av}-value obtained for these samples. This can be explained by the fact that as the grain size decreases, the number of grain boundaries that photons of light encounter on their way to the photoreceptor increases. This leads to an increase in the influence of light scattering, as well as a complication of

the trajectory of a light photon and an increase in the length of its path, which, in turn, increases the probability of absorption of this light photon.

For polycrystalline scintillators, both in the case of stilbene and in the case of *p*-terphenyl, it cannot be asserted that there is any dependence of the value of *T* on the average size L_{av} of grains. Such results can be explained as follows. During the manufacture of composite scintillators, the crystal grains and their size do not undergo any changes. During the production of polycrystals by the hot pressing, processes of recrystallization, sintering, as well as cracking of crystalline grains can occur, which can lead to a change in the average size L_{av} of grains. It seems logical to assume that large-sized grains have a higher probability of cracking compared to small-sized grains, which will cause a decrease in their average size L_{av}. Conversely, for small-sized grains, the sintering process should occur more easily, compared to large-sized grains, which should cause an increase in L_{av}-value. As a result, the average size L_{av} of grains in the obtained polycrystals can be somewhat "evened out" for samples obtained from grains of different fractions. However, the verification of this assumption requires a separate study.

To determine the ability of organic composite scintillators to separate detection of ionizing radiations of different types by the charge-time transformation method we have obtained the samples with a diameter of d = 20 mm and a thickness of h = 5 mm based on stilbene and *p*-terphenyl grains.

Tables 1 and 2 present the *FOM*-values for stilbene composite detectors and p-terphenyl composite detectors, respectively. The *FOM*-values for the reference single crystals are presented as well.

Table 1

FOM-values for the single crystal and composite scintillators of stilbene

Fraction of grains, mm	*FOM*
Single crystal	2.10
0.1…0.3	1.53
0.3…0.5	1.65
0.5…1.0	1.58
1.0…1.5	1.53
1.5…2.0	1.73

Table 2

FOM-values for the single crystal and composite scintillators of p-terphenyl

Fraction of grains, mm	*FOM*
Single crystal	1.45
0.1…0.3	1.03
0.3…0.5	0.84
0.5…1.0	1.11
1.0…1.5	1.07
1.5…2.0	1.08

Evaluating the data in Tables 1 and 2, it can be concluded that for composite samples based on both stilbene and *p*-terphenyl, the *FOM*-values, firstly, do not have a certain dependence on the size of the grains of the scintillating substance (in the studied range). Secondly, composite samples have sufficiently high *FOM*-values in comparison with the single crystals: for stilbene composite detectors it is on average 76% of the corresponding single crystal and for *p*-terphenyl composite scintillators it is 70% of the corresponding single crystal. Besides, the *FOM*-values for the samples based on *p*-terphenyl are about 70% of the *FOM*-values for the samples based on stilbene.

In general, the obtained results indicate that for organic polycrystalline and composite scintillators under consideration, as well as for organic single crystals, standard methods of measuring the light output, the optical transmission coefficient, and the *FOM*-value by the charge-time transformation method can be applied.

The research of luminescence spectra at this stage of the work was carried out as a control and evaluation of the purity of the scintillation material, which was synthesized and purified during the study process. Control took place after the creation of experimental samples: growth and processing of a single crystal ingot, pressing of a polycrystalline scintillator, and polymerization of a composite scintillator. The luminescence spectra showed characteristic luminescence lines of organic samples of single crystal, polycrystalline, and composite scintillators based on *p*-terphenyl and stilbene at 390 and 420 nm, respectively. It should be noted that there were additional lines in the luminescence spectrum characterizing other impurities in scintillation materials. However, their intensity was much lower than the maximum luminescence intensity of the main luminescence lines (~2…3%), that is, their contribution is insignificant. Thus, it can be stated that the synthesized and/or purified scintillation substances are sufficiently pure and meet the required quality.

CONCLUSIONS

1. In the case of excitation by alpha particles, composite and polycrystalline scintillators based on stilbene grains show similar dependences of the value of the relative light output *J*, as well as the energy resolution *R* on the average grain size L_{av}. Initially, with an increase in the L_{av}-value (approximately 0.8 mm), there is a significant increase in the *J*-value and improvement in the *R*-value of the samples. For L_{av}-values greater than 0.8 mm, the light output *J* of the samples, as well as their energy resolution *R* do not change significantly.

2. For composite scintillators based on *p*-terphenyl, the dependences of the relative light output *J* and energy resolution *R* on the average grain size L_{av} were observed similar to scintillators based on stilbene grains. For polycrystalline scintillators based on *p*-terphenyl we did not observed the dependence of the *J*-value and the *R*-value on the average grain size L_{av}. Such results may be related to the fact that the sintering and recrystallization processes occurring during the hot pressing process proceed differently in stilbene and *p*-terphenyl, which requires further research.

3. For composite samples based on both stilbene and *p*-terphenyl, the *FOM*-values, firstly, do not have a certain dependence on the size of the grains (in the studied range). Secondly, composite samples have

sufficiently high *FOM*-values in comparison with the single crystals: for stilbene composite detectors it is on average 76% of the corresponding single crystal and for *p*-terphenyl composite scintillators it is about 70%.

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ГЕТЕРОСТРУКТУРОВАНІ СЦИНТИЛЯТОРИ НА ОСНОВІ ОРГАНІЧНИХ МОНОКРИСТАЛІЧНИХ ГРАНУЛ ДЛЯ РОЗДІЛЬНОЇ РЕЄСТРАЦІЇ ІОНІЗУЮЧИХ ВИПРОМІНЮВАНЬ ЗА ФОРМОЮ ІМПУЛЬСУ

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Вивчено властивості та вдосконалено характеристики нових типів органічних гетероструктурованих сцинтиляторів як детекторів швидких нейтронів і короткопробіжних заряджених частинок. Ці дослідження спрямовані на пошук можливостей покращення характеристик детектора на основі гетероструктурованого сцинтиляційного матеріалу та розширення можливостей його практичного застосування, зокрема у задачах роздільної реєстрації іонізуючих випромінювань різних типів.