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ORGANIC POLYCRYSTALS WITH IMPROVED OPTICAL PROPERTIES

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Polycrystalline scintillators are promising materials for solving a number of specific problems. However, their disadvantage is that the optical characteristics significantly deteriorate with increasing thickness of the scintillator. This study examines the scintillation and optical properties of polycrystals made from stilbene and *p*-terphenyl of various thicknesses. Of all the materials used, stilbene plates obtained by recrystallization from an organic solvent are the best and the most promising for obtaining polycrystals with the best scintillation and optical characteristics.

PACS: 32.50.+d; 78.60.−b; 87.53

INTRODUCTION

The priority scientific focus is the creation of cutting-edge scintillation materials and detectors based on them to address a wide range of modern problems related to scintillation materials and devices. This includes the development of detectors for high-energy physics experiments and solutions to environmental radioecology problems.

Increasing the light output of the scintillator improves the reliability of the detector's operation. This is particularly important for low-energy radiation detection tasks, when the signal amplitude becomes comparable to the amplitude of noise pulses measured by the equipment. However, a significant disadvantage of organic polycrystalline scintillators is that their scintillation characteristics deteriorate significantly with increasing thickness [1].

Methods are currently known for obtaining polycrystalline organic scintillators by either cold [2–4] or hot [1, 5–10] pressing of crystalline granules. These granules can be obtained by crushing a previously grown single crystal or a crystalline ingot formed during the purification of the initial material by zone melting [9]. Polycrystals can also be produced by pressing plates obtained by the recrystallization of raw material in an organic solvent [11, 12]. Another method involves obtaining a *p*-terphenyl polycrystals from fragments of an ingot obtained after the reverse movement of the zone purification ampoule [13]. While material for polycrystals production in granule form is prevalent in most studies, material for polycrystals production from plates has received very little attention. The largest number of studies is devoted to investigating how the size of the granules affects the characteristics of polycrystalline scintillators. At the same time, the majority of studies focus on thin polycrystals with a thickness of no more than 5 mm. Notable works related to the study of thicker polycrystals include [1, 7]. However, these studies are limited to the use of *p*-terphenyl granules [1] or stilbene [7] for the production of polycrystals.

The work aims to study and compare the scintillation and optical properties of a series of polycrystals of various thicknesses made from stilbene and *p*-terphenyl granules and plates.

1. EXPERIMENTAL

1.1. OBTAINING OF POLYCRYSTALLINE SAMPLES

The material for obtaining granules of stilbene and *p*-terphenyl was fragments of a single crystal obtained by the Bridgman method. Single crystal ingots were placed in a fluoroplastic mortar, to which liquid nitrogen was added. The cryogenic crushing method minimizes mechanical loads that negatively affect the structural perfection of granules of molecular organic substances, reducing their transparency. From the granules obtained as a result of crushing, a fraction of granules of stilbene (a) and *p*-terphenyl (b), 2.5…3.0 mm in size, was isolated using calibrated sieves.

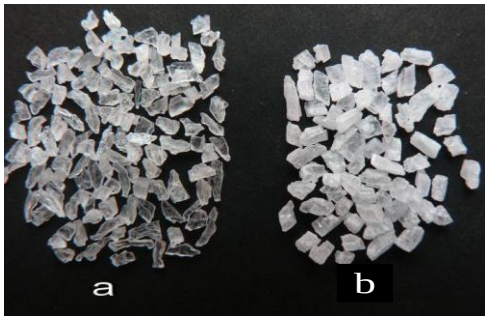


Fig. 1. Granules 2.5…3.0 mm in size obtained after crushing a single-crystal ingot of stilbene (a) and p-terphenyl (b)

The material for obtaining the plates was synthesized stilbene powder and *p*-terphenyl powder purchased from ACROS ORGANICS BVBA. To obtain the material in the form of plates, stilbene and *p*-terphenyl powders were recrystallized in 1,2-dichloroethane (Fig. 2).

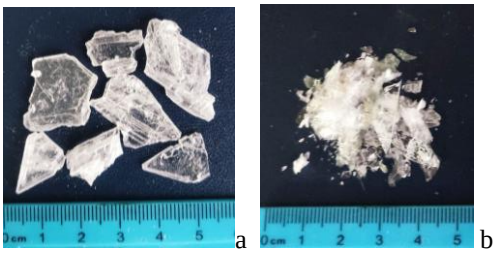


Fig. 2. Images of stilbene (a) and p-terphenyl (b) plates obtained after recrystallisation

The thickness of the stilbene plates measured with a micrometer was 1.0…2.5 mm, the thickness of the *p*-terphenyl plates measured with a microscope was 50 μm.

To make polycrystals, a weight of starting material corresponding to the thickness of the polycrystal to be obtained was poured into a 20 mm diameter mold, which was inserted into a heater and placed under a hydraulic press. The temperature was gradually raised to the pressing temperature over the course of one hour. For the stilbene polycrystal, the pressing temperature was (100±5) °C, and for *p*-terphenyl, it was (160±5) °C. Pressing was carried out for one hour. A press force equal to one ton was applied, which for a punch diameter of 20 mm corresponded to a pressure of 30 MPa.

1.2. EXPERIMENTAL METHODS

The relative light output of polycrystalline scintillators was determined by the standard method using scintillation amplitude spectra. The photomultiplier 9208A was used as a photodetector [11]. The amplitude distribution of scintillation pulses was obtained using an amplitude-to-digital converter. The value of the relative light output L was calculated according to (1)

$$L = \left(J / J_{ref} \right) \times 100\ \%, \qquad (1)$$

where J is the value of the amplitude that corresponds to the center of gravity of the peak in the spectrum of the scintillator under investigation, J_{ref} is the value of the amplitude that corresponds to the center of gravity of the peak in the spectrum of the reference scintillator. The single crystal of *p*-terphenyl was used as a reference detector. We used the following types of ionizing radiations: alpha particles from ^{239}Pu with energy E_α = 5.15 MeV, conversion electrons from ^{137}Cs with energy E_β = 0.622 MeV.

The optical transmittance of the samples was determined using a Shimadzu UV 2450 spectrophotometer [12]. We calculated the value of optical transmittance T as follows:

$$T = \left(L / L_0 \right) \times 100\ \%, \qquad (2)$$

where L_0 is the flux of light falling on the sample, L is the flux of light that propagates through the sample. The T-value (2) is the relative light transmittance, where T = 100% is the light transmittance of air at the room temperature.

2. RESULTS OF STUDIES AND THEIR ANALYSIS

For the research, four series of polycrystals (Fig. 3) with a diameter of d = 20 mm and a thickness of 2, 3, 4, 5, 7, and 10 mm were obtained, where *p*-terphenyl and stilbene granules, *p*-terphenyl and stilbene plates were used as the material for pressing.

The relative light output L when alpha particles (^{239}Pu), the energy resolution R, excited the samples and the optical transmission coefficient T were measured. The amplitude scintillation spectra of organic scintillators samples were measured when excited by alpha particles, the energy resolution R of the corresponding peaks (the ratio of the peak maximum position to the peak width at half height, expressed as a percentage) and the optical transmission T of the samples are standard procedures. Typical results of such measurements can be found, for example, in [1, 5]. Therefore, this article presents only the final results based on the analysis of the original experimental data.

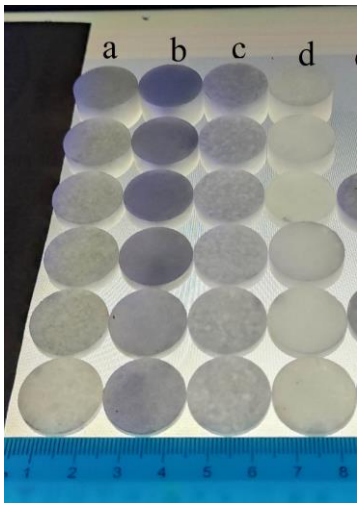


Fig. 3. Images of p-terphenyl and stilbene polycrystals with thicknesses of 2, 3, 4, 5, 7, and 10 mm (from bottom to top): a – p-terphenyl PC obtained from granules; b – p-terphenyl PC obtained from plates; c – stilbene PC obtained from granules; d – PC of stilbene obtained from plates

For an alpha particle, the path length in *p*-terphenyl is approximately 30 μm. This means that photons of light are created on the surface of the scintillator and must travel through the entire thickness of the sample before they can be recorded. Therefore, light scattering processes have a stronger effect on the values of L and R in the case of alpha particle excitation. In the case of alpha particle excitation, the optical transmission values should correlate with the light output values.

The amplitude scintillation spectra of the studied samples obtained by irradiation with alpha particles (^{239}Pu) are shown in Figs. 4.1–4.4. A 5 mm thick stilbene single crystal was used as a reference.

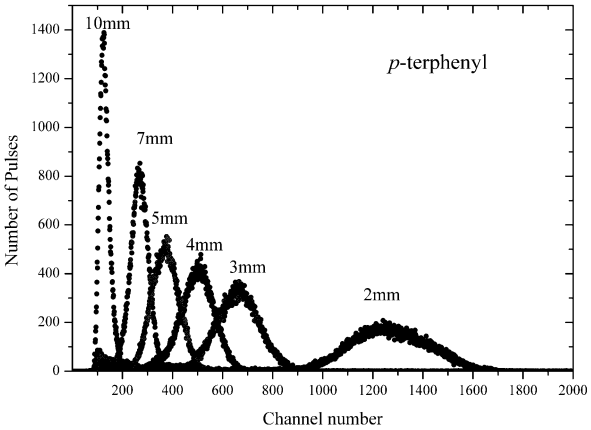


Fig. 4.1. The amplitude scintillation spectra of the p-terphenyl polycrystals made from granules and the reference stilbene single crystal with h of 5 mm upon alpha-particles irradiation (^{239}Pu). The numbers indicated in the figure show the thickness of the sample for which the corresponding spectrum was obtained

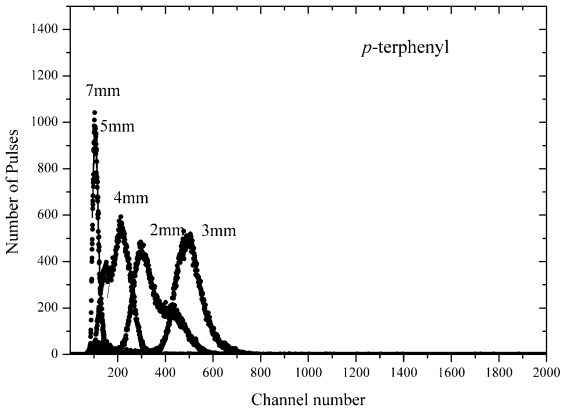


Fig. 4.2. The amplitude scintillation spectra of the p-terphenyl polycrystals made from plates and the reference stilbene single crystal with h of 5 mm upon alpha-particles irradiation (^{239}Pu). The numbers indicated in the figure show the thickness of the sample for which the corresponding spectrum was obtained

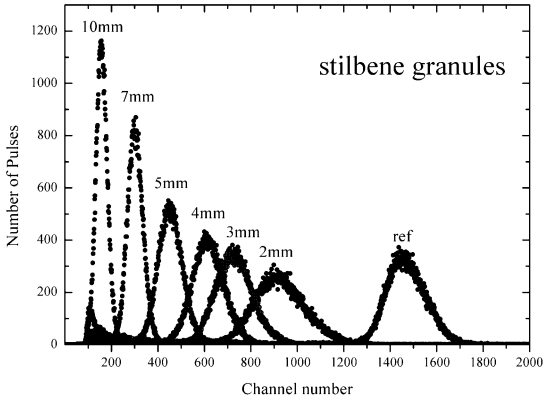


Fig. 4.3. The amplitude scintillation spectra of the stilbene polycrystals made from granules and the reference stilbene single crystal with h of 5 mm upon alpha-particles irradiation (^{239}Pu). The numbers indicated in the figure show the thickness of the sample for which the corresponding spectrum was obtained

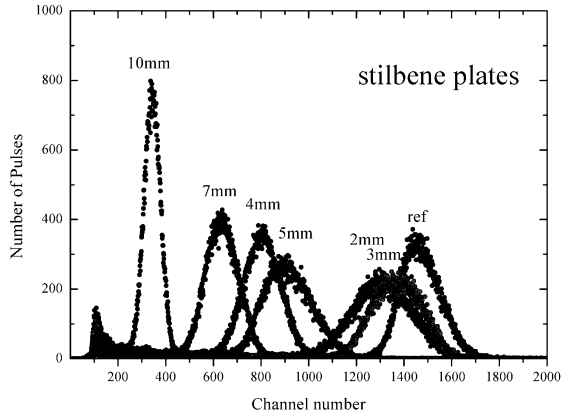


Fig. 4.4. The amplitude scintillation spectra of the stilbene polycrystals made from plates and the reference stilbene single crystal with h of 5 mm upon alpha-particles irradiation (^{239}Pu). The numbers indicated in the figure show the thickness of the sample for which the corresponding spectrum was obtained

Fig. 5 presents the results of optical transmission measurements for samples *p*-terphenyl and stilbene polycrystals made from granules and plates obtained from different starting materials of 5 mm thicknesses.

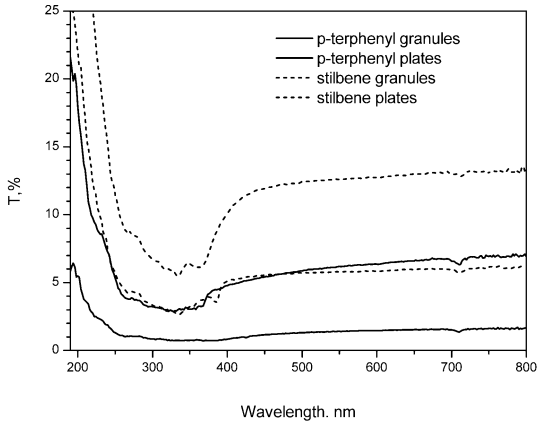


Fig. 5. Optical transmittance values for p-terphenyl and stilbene polycrystals made from granules and plates obtained from different starting materials of 5 mm thicknesses

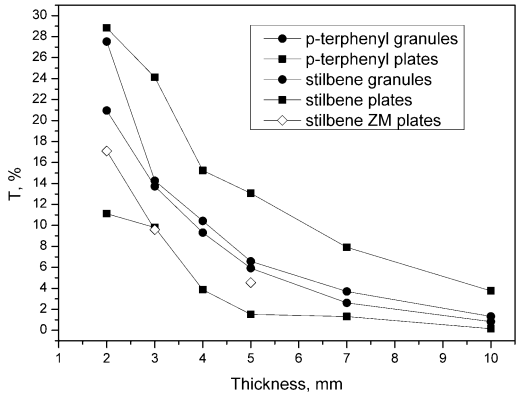


Fig. 6. Optical transmittance values at a wavelength of 700 nm for p-terphenyl and stilbene polycrystals made from granules and plates of different thicknesses obtained from different materials

Fig. 6 presents a comparison of the optical transmittance at 700 nm for *p*-terphenyl and stilbene polycrystals, prepared from granules and plates derived from different starting materials, with a thickness of 5 mm.

The values of relative light output *L*, energy resolution *R* and optical transmittance *T* are given in Table.

Despite the apparent difference in transparency of the cryogenically crushed granules of stilbene and *p*-terphenyl (see Fig. 1), the optical transmittance values of the polycrystals obtained from them are practically the same. The use of plates obtained by recrystallization from 1,2-dichloroethane as a starting material for obtaining polycrystals is positive in the case of stilbene and negative in the case of *p*-terphenyl. This is due to the thickness of the plates obtained by recrystallization. As in the case of studies of the influence of the size of the granules on the optical properties of polycrystals [6, 7], the maximum optical transmittance values were observed for the material with a greater thickness. The possibility of obtaining plates of greater thickness for stilbene and the impossibility for *p*-terphenyl is explained by the different crystal structure. The herringbone crystal structure of stilbene is characterized by the production of

thick plates, while the π-folded crystal structure of *p*-terphenyl makes it difficult to obtain thicker plates by recrystallization from commonly available organic solvents.

Comparison of relative light output L, energy resolution R and optical transmission coefficients T

Sample	Thickness, mm	*L*, % ^{239}Pu	*R*, %	*T*, %	
				λ =390 nm	λ = 700 nm
Stilbene single crystal (reference)	5	100	11.7	–	–
p-terphenyl polycrystals obtained from granules	2	86.3	23.8	23.70	27.53
--//--	3	46.1	28.0	11.14	14.53
--//--	4	35.5	24.6	7.37	10.42
--//--	5	26.0	31.7	4.52	6.58
--//--	7	18.6	28.9	2.27	3.70
--//--	10	8.5	35.2	0.54	1.32
p-terphenyl polycrystals obtained from plates	2	20.3	33.2	8.40	11.16
--//--	3	33.0	24.1	7.64	9.79
--//--	4	14.7	50.2	2.08	3.86
--//--	5	10.3	26.5	0.77	1.52
--//--	7	7.3	28.1	0.51	1.31
--//--	10	–	–	0.03	0.15
stilbene polycrystals obtained from granules	2	62.5	20.4	18.01	20.95
--//--	3	50.0	23.4	11.41	13.72
--//--	4	41.6	22.3	7.11	9.31
--//--	5	30.8	25.2	4.40	5.92
--//--	7	21.0	23.3	1.77	2.62
--//--	10	10.7	37.3	0.47	0.83
stilbene polycrystals obtained from plates	2	90.2	16.2	21.43	28.83
--//--	3	96.4	15.9	19.36	24.12
--//--	4	54.8	20.8	10.96	15.24
--//--	5	63.4	23.8	9.27	13.07
--//--	7	44.2	24.0	5.96	7.91
--//--	10	23.2	24.0	2.07	3.76

CONCLUSIONS

1. Use of plates obtained by recrystallization from 1,2-dichloroethane as starting material for obtaining polycrystals: positive in the case of stilbene and negative in the case of *p*-terphenyl.

2. The scintillation and optical characteristics of stilbene and *p*-terphenyl polycrystals obtained using granules as the starting material do not have significant differences.

3. For stilbene polycrystals obtained from plates, it has been shown that the light yield values relative to the reference stilbene single crystal are over 60% when excited by alpha particles. For polycrystals made from stilbene and *p*-terphenyl granules, this value is up to 30%.

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ОРГАНІЧНІ ПОЛІКРИСТАЛИ З ПОКРАЩЕНИМИ ОПТИЧНИМИ ВЛАСТИВОСТЯМИ

І.В. Лазарєв, Я.І. Полупан, А.В. Креч, І.Ф. Хромюк, Є.В. Мартиненко, С.В. Махота, С.С. Міненко

Полікристалічні сцинтилятори є перспективними матеріалами для вирішення низки специфічних проблем. Однак їхнім недоліком є те, що оптичні характеристики значно погіршуються зі збільшенням товщини сцинтилятора. У цьому дослідженні розглядаються сцинтиляційні та оптичні властивості полікристалів, виготовлених зі стильбену та *n*-терфенілу різної товщини. З усіх використаних матеріалів пластини стильбену, які отримані перекристалізацією з органічного розчинника, є найкращими та найперспективнішими для отримання полікристалів з найкращими сцинтиляційними та оптичними характеристиками.