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Influence of manufacturing conditions of detectors based on crystals of SrI_2:Eu on their scintillation characteristics

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The factors determining the characteristics of the SrI_2:Eu scintillator during processing and packing stages are considered. Using experimental and computer modeling, we selected options for processing the surfaces of scintillators, providing an improvement in their scintillation characteristics. It is shown that the method of processing the surface of crystal Sr_2:Eu size of ∅21×5 mm and ∅21×10 mm grown by method of synthesis of raw materials components, combined with the diffuse reflector Tetratex provides improved energy resolution of 3.65 % and 3.87 % respectively at 662 keV.

Keywords: Scintillator, light output, energy resolution.

Рассмотрены факторы, определяющие характеристики сцинтилляторов $SrI_2:Eu^{2+}$ на стадии их обработки и упаковки. С помощью экспериментального и компьютерного моделирования выбраны варианты обработки поверхностей сцинтилляторов, обеспечивающие улучшение их сцинтилляционных характеристик. Показано, что способ обработки поверхности кристаллов SrI_2:Eu размерами ∅21×5 мм и ∅21×10 мм, выращенных методом синтеза исходных компонентов сырья, в сочетании с диффузным отражателем Tetratex, улучшает разрешающую способности детектора для энергии γ-квантов 662 кэВ до 3,65 % и 3,87 % соответственно.

Вплив умов виготовлення детекторов на основі кристалів SrI_2:Eu на їх сцинтиляційні характеристики. *В.А.Тарасов, Т.Є.Горбачева, Н.В.Реброва, А.А.Бобовніков, Є.П.Галенін, Л.А.Андрющенко, С.Д.Светлічна.*

Розглянуто чинники, що визначають характеристики сцинтиляторів $SrI_2:Eu^{2+}$ на стадії їх обробки і упаковки. За допомогою експериментального і комп'ютерного моделювання обрані варіанти обробки поверхонь сцинтиляторів, що забезпечують поліпшення їх сцинтиляційних характеристик. Показано, що спосіб обробки поверхні кристалів SrI_2:Eu розмірами ∅21×5 мм і ∅21×10 мм, вирощених методом синтезу вихідних компонентів сировини, у поєднанні з дифузним рефлектором Tetratex, покращує роздільну здатність детектора для енергії γ-квантів 662 кеВ до 3,65 % і 3,87 % відповідно.

1. Introduction

The most widely used gamma detectors for radiation monitoring are the alkali halide scintillators NaI:Tl [1, 2]. The best energy resolution achievable on such materials is 5.8 %, and the light output of 45000 ph/MeV for 662 keV gamma-rays from the ^{137}Cs source [3].

The creation of new instruments for radiation monitoring and radiation safety requires continuous improvement of scintillation detectors. Efforts for improving of scintillation detectors characteristics are targeted on creation of new materials and on development of special designs of detectors and methods of their manufacture [4].

Recently, a number of effective alkaline-earth scintillation materials have been obtained that have high scintillation characteristics, among which a special place is taken by the strontium iodide scintillator doped with divalent europium $SrI_2:Eu^{2+}$. The high atomic number (48) and density (4.6 g/cm^3), a good resolution capability make it a promising scintillator material for radiation control purposes. The prospect of application of $SrI_2:Eu$ when designing a submersible double-crystal gamma spectrometry to monitor the aqueous medium is demonstrated in [5]. Low intrinsic background [6, 7], perfect matching of scintillation spectrum with spectral sensitivity of silicon photomultipliers (SiPM), a high degree of light output proportionality and high radiation hardness provided the possibility of application of the $SrI_2:Eu$ scintillator in a gamma spectrometer for determination of the chemical composition of an asteroid [8]. The light output and energy resolution of small samples of $SrI_2:Eu$ scintilltors obtained in different laboratories varies over a wide range. In particular, their light output varies from 68000 ph/MeV to 120,000 ph/MeV [9, 11, 13]. Energy resolution varies over a wide range from 2.6 % to 5.5 % and more [9–11, 13–16] which depends on a number of factors.

The main negative feature of $SrI_2:Eu$ crystals is its low transparency to its own radiation which leads to strong scintillation light reabsorption and consequently to reduction in light output and resolution of the scintillator with increasing size [9, 10]. Additionally, high hygroscopicity and reactivity of crystals creates problems during growing. To obtain crystals with high scintillation parameters a high concentration of europium is used; therefore, the probability of the formation of complexes containing oxygen compounds increases.

A critical parameter for the mass production and application of $SrI_2:Eu$ scintillators is the high cost of the raw material for growing. Minimum purity of raw material for the preparation of superior grade scintillators with resolution of better than 4 % at 662 keV ^{137}Cs should be higher than 4N. Furthermore, such $SrI_2:Eu$ scintillation crystals require ultra dry SrI_2 and EuI_2 salts [11] which have high cost ($1000 kg). In [12], an inexpensive method for the synthesis of the starting components of raw materials for growing $SrI_2:Eu$ crystals was proposed. Special technologies for dehydration of the raw material and multiple recrystallization can reduce the concentration of oxygen-containing impurities in $SrI_2:Eu$ crystals. $SrI_2:Eu$ crystals with a diameter of up to 26 mm and a length of up to 70 mm having an energy resolution of 4.4 % upon excitation by a source of ^{137}Cs were grown from the obtained raw materials.

The aim of this work is to study the possibility of improving performance of scintillation detectors based $SrI_2:Eu^{2+}$ crystals, grown by method described in [12] by special surface treatment and packing.

2. Factors that determine the characteristics of $SrI_2:Eu$ scintillators at various stages of their manufacture

The quality of scintillators to a decisive degree depends on the technology of single crystals growing. Scintillation characteristics of crystals depend on both intrinsic and impurity caused point lattice defects [17]. Scintillation characteristics may deteriorate depending on the presence of certain anionic radicals in the bulk and on the degree of hydration of the surface. The effect of impurity ions on the characteristics of $SrI_2:Eu$ crystals was studied in [18]. In this work, it was shown that for $SrI_2:Eu$ crystals, the main impurity which significantly impairs the scintillation characteristics, is the OH^- anion. For the growth of high-quality crystals with improved scintillation characteristics, it is preferable to use raw materials with pH < 3–4.

The quality of detectors is determined by conditions of mechanical processing and packaging [19, 20] in particular by the state of surface of active elements. The average value of the light path depends on the nature of the surface of the scintillator, which affects the light output and the scatter of the light paths from the origin to the photodetector. This in turn affects the energy resolution. The surface state of the scintillator also affects the stability of its characteristics during operation of the detector.

The surface condition of the scintillator is determined by both the properties of the material being processed and the processing conditions. Extremely high hygroscopic crystals SrI_2:Eu is taken into account at all stages of the manufacturing process of the detector. All operations are performed in "dry" areas or boxes. Deteriorated layer (DL) on the crystal surface is formed during mechanical processing of the surface of the crystals. Proper selection of techniques and modes of processing gives the opportunity to receive the minimum value of the DL and improve the performance of the scintillatior. The quality of scintillator surface treatment by abrasives depends on the choice of abrasive size distribution and structure and on a method of removing of the residue with abrasive from machined surface [20]. Cutting crystals should be carried out under a layer of mineral oil [22] since it has hydrophobic properties and prevents the substance from contacting with air. Mineral oil is also used as the wetting liquid during polishing.

Despite the fact that the surface treatment of initial crystalline material and packaging of ready scintillators is carried out in an inert atmosphere or an atmosphere of dry air a hydrolysis film is formed on their surface. During machining, it is not possible to completely remove bound water from the surface and in the sub surface layer. The presence of residual moisture creates conditions for the hydrolysis of the material (especially under the influence of light), which is accompanied by the formation of iodine condensate and the degradation of scintillation parameters. Any traces of moisture or the presence of hydrated zones on the surface of a packed scintillator over time leads to irreversible changes in its optical and scintillation characteristics.

To solve this problem, a method for treating of Eu-containing crystals characterized by high hygroscopicity and chemical activity, in particular, LiI:Eu is proposed in [23]. In this method, hexamethyldisilazane (HMDS) is used as a wetting liquid for grinding the surface of crystals and cleaning the treated surface. The use of HMDS as an active washing liquid allows not only to clean the surface of crystals from traces of abrasive material, but also to reduce the residual moisture content in the surface layers, which reduces the likelihood of hydrolysis of the scintillator material [24].

Scintillators for practical applications are packaged in sealed containers. The need for careful packing of hygroscopic crystals is noted in [16]. In this work, a series of experiments with different packaging options was carried out. For SrI_2:Eu crystals with a volume of less than 1 cm^3, the resolution better than 2.7 % at 662 keV was obtained and for a crystal with a volume of 3 cm^3 ~ 3 %. It is shown that important factors in the manufacture of detectors are: scintillator geometry, method of processing, reflecting material and material of the output optical window. The advantage of using Teflon diffuse reflective material in comparison to specular reflector is noted. The authors found that the use of high-quality polishing of the scintillator surface with a suspension of diamond powder with a grain size of 3 μm in mineral oil, the use of Teflon diffuse reflectors, a thin window, and optical adhesive composition to create optical contact between the scintillator and the detector output window provide improved light collection conditions. To verify the uniformity of light collection different regions of the crystal were irradiated using a collimator. It turned out that by using the correct packaging it is possible to reduce the scatter in the light output of the scintillator for its upper and lower parts to less than 1 %.

Typically, the shape and size of scintillator are determined by experimental technical problem solved using scintillation spectrometer. The choice of a method for treating scintillator surfaces that provides a given degree of roughness should provide optimal conditions for light collection.

Below are the results of studies aimed at improving the scintillation characteristics of SrI_2:Eu crystals by choosing the option of their surfaces processing.

3. Computer simulation of the choice of surface treatment of SrI_2:Eu scintillators

Computational methods are commonly used to investigate the light-collection conditions in scintillators. The most popular one is a computer simulation using a Monte-Carlo method.

In the present work, a scintillator based on a SrI_2:Eu single crystal with dimensions of 21×5 mm was used as an object for computer simulation. A crystal with the indicated dimensions was also used for experimental studies.

A significant effect on the magnitude of the light output is exerted by the type of external reflective material. The most commonly used is a diffuse reflector having a

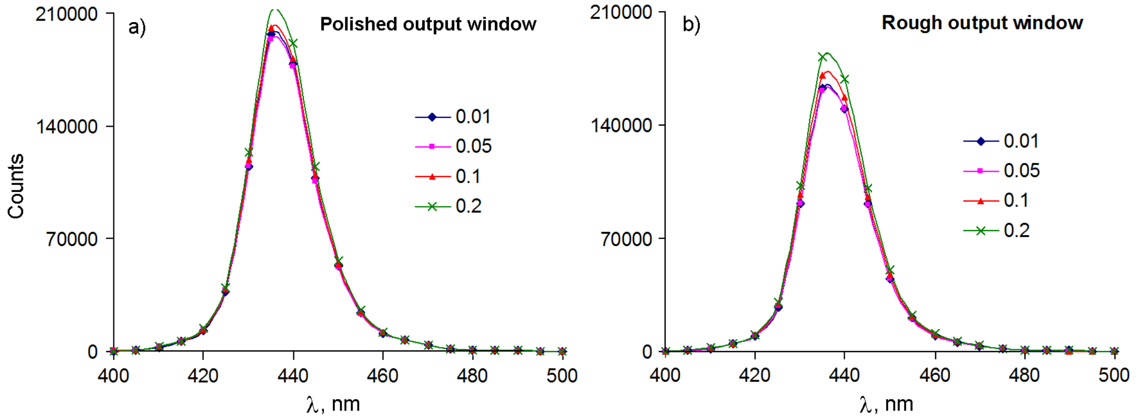

Fig. 1. Simulated scintillation spectra for different surface treatments of the SrI_2:Eu scintillator with a size of ∅21×5 mm when polishing (a) and grinding (b) the output window.

reflection coefficient of 0.95 which provides a high light output for scintillators with varying degrees of roughness. In computer modeling the type and reflection coefficient, the number of emitted photons remained unchanged and the size and shape in the form of a cylinder were varied.

Two types of scintillator output window processing were considered: rough polishing with conventional roughness value of 0.05 — which corresponds to the F1000 type of abrasive processing (grain size of the main fraction of 3–5 mm); finishing polishing with a parameter of 0.01 (corresponds to processing with an abrasive like M 1 with a grain size of 1 μm or less). The lateral and upper inlet end surfaces were treated identically.

For each type of processing of the output window light collection was simulated for four types of processing of the side and top surfaces with conditional parameters: 0.01 — polishing ; 0.05 — fine grinding; 0.1 ; 0.2 — grinding. A 1000 flashes were considered in each simulation with equal probability distributed over the whole volume of the scintillator. From each flash generation site 1000 photons were emitted in 4π with an equal probability.

In Fig. 1 simulated scintillation spectra are presented for different types of surface treatment of the SrI_2:Eu scintillator with dimensions of ∅21×5 mm with polished (a) and grinded (b) output window.

It can be seen that with a change in the processing of the upper entrance surfaces and side surfaces (parameters 0.01; 0.05; 0.1 and 0.2), the scintillation intensity changes slightly and increases with an increase in the roughness of these surfaces (from parameter 0.01 to 0.2). The intensity of scintillations varies more significantly during the transition from the polished surface of an output window (Fig. 1a) to the ground surface (Fig. 1b).

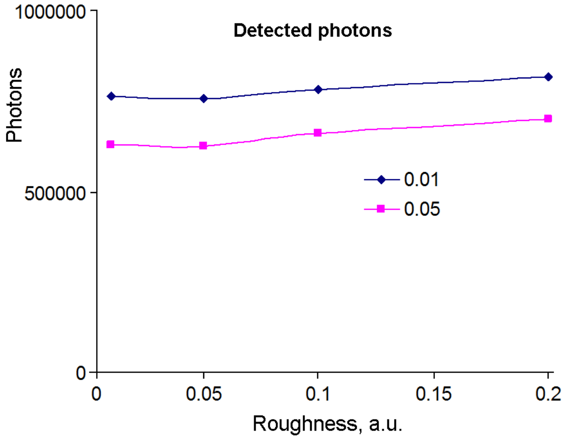

Fig. 2. Change in the scintillation intensity from the degree of roughness of the surface of the output window (parameters 0.01 and 0.05) and the roughness of other surfaces of the scintillator (parameters on the abscissa axis).

These changes in the intensity of scintillations, in the form of the dependence of the number of registered photons on the degree of surface roughness, are shown in Fig. 2.

It is seen that in the case of the polished surface of the output window (parameter 0.05), the number of registered photons decreases. This is due to an increase in the average path of photons to the output window and an increase in the probability of their loss along this path, which is reflected in Fig. 3.

Thus the results of the simulation clearly indicate the need for polishing of output window of scintillator and grounding of side and top parts of its surface.

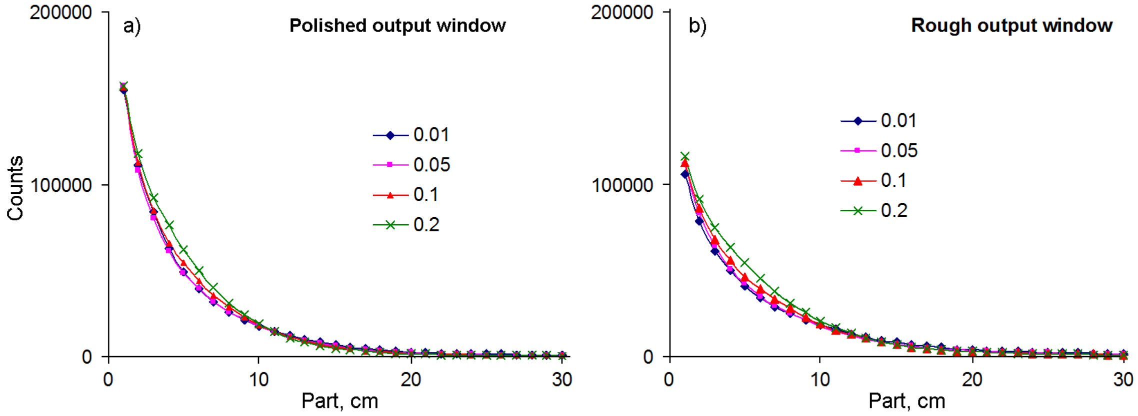

Fig. 3. Dependence of the average path of photons to the output window on the roughness of the surface of the output window (parameters 0.01 and 0.05) and the roughness of other surfaces of the scintillator (parameters on the abscissa axis).

The processing option with grinding of the side and upper end surfaces (input window) and with polishing of the lower end surface (output window) was experimentally tested.

4. Experiment

Method based on using HMDS proposed in [23] was modified processing of $\mathrm{SrI_2}$:Eu scintillators. The need to modify the process due to the fact that after grinding with abrasive powder in composition with HMDS of the exit window the optical properties of its surface become deteriorated. Final processing using LiF powder with a size of abrasive grains of 3–5 mm does not allow to completely eliminate DL formed during grinding. This leads to a decrease in the optical properties of the output surface of the scintillator and consequently to a deterioration in its scintillation characteristics. In addition, the use of alumina powder with an abrasive size of 63 μm for matting the inlet and side parts of the scintillator surface leads to the appearance of deep scratches with the formation of nuclei of centers of recrystallization. With repeated grinding, this leads to the loss of blocks from the surface layer and a decrease in scintillation characteristics.

During developing of modified process the results of the studies presented in [20, 25, 26], aimed at minimizing the DL by using superfine powders at the finish polishing step. In modified method after processing of crystal surfaces and washing them using HMDS dry processing was sequentially performed to remove HMDS effect. Output surface of the scintillator was additionaly polished by the abrasive with grains of 0.3–0.6 μm. Finish processing of the remaining surfaces was done with the use of polymeric 3M TM Imperial TM films containing an abrasive powder with the size of grain of abrasive main fraction of 15–30 μm, which with a polymeric binder is clipped to the polyester substrate. The special technology of applying abrasive grains to the polymer base ensures their identical orientation [27]. It is known that application of chosen abrasive allows improving the quality of the processed organic [28] and inorganic crystals [29].

As objects for experimental studies, blanks of $\mathrm{SrI_2}$:Eu scintillators with a europium concentration of 5 % were used. This concentration of dopant is optimal to achieve high scintillation parameters presented in [11, 14].

Surface treatment of $\mathrm{SrI_2}$:Eu single crystals (No.1–3) sized ∅21×5 mm (No.5–7) sized ∅21×10 mm (number No.1–3) and ∅21×5 mm (5–7) was carried out by a modified method, and $\mathrm{SrI_2}$:Eu crystals with dimensions of ∅21×10 mm (4) and ∅21×5 mm (8) in accordance with the technical solution [23].

The surface treatment of the scintillator blanks was carried out in a "dry" room. At different stages of the treatment, the spectrum of pulse amplitudes in $\mathrm{SrI_2}$:Eu scintillators was measured directly in the "dry" room using gamma radiation with an energy of 662 keV from a ^{137}Cs source. A photomultiplier tube PMT R1307 from Hamamatsu was used as a photodetector.

Prior to measurement crystals were wrapped into diffuse reflector based on fluoroplastic film Tetratex TX3104 PTFE.

Table 1. Scintillation characteristics of ∅21×5 mm SrI_2:Eu detectors

Specimen No.	Processing stage	Sequence of operations	Resolution, %
1	1	1. Grinding all surfaces with F 400 powder with an abrasive grain size of 20 μm in the composition with HMDS. 2. Flushing the polished surface of the HMDS	4.25
2	–	–	4.29
3	–	–	4.20
	2	For all samples : 3. Grinding with successive reduction in particle size from 14 to 6 microns. 4. Flushing the polished surface of the HMDS . 5. Sequential polishing of scintillator surfaces on the output window side with LiF powder with a size of 5 microns and 3 microns and then wash with HMDS. 9. Processing of the side and entrance surfaces with 3M ^{TM} Imperial ^{TM} polymer film with the grain size of the main fraction:	
1	–	15 μm	3.80
2	–	20 μm	3.83
3	–	25 μm	3.78
1	3	Dry polishing of the scintillator surface from the side of the output window with 0.3 μm LiF powder	3.66
3	–	–	3.65
2	–	Dry scintillator polishing of the output window surface with LiF powder with a size of 0,6 μm	3.67

5. Results and discussion

Table 1 shows a sequence of processing operations of SrI_2:Eu scintillators surfaces with dimensions of ∅21×5 mm and measured energy resolution at different stages of their processing.

Fig. 4 illustrates the change in the pulse-height spectra at various stages of processing the surfaces of the SrI_2:Eu scintillator size ∅21×5 m (specimen No. 3).

From the analysis of the results given in Table 1 and Fig. 4 it follows that an improvement in the light output and resolution of the specimens is achieved by reducing the degree of roughness on the side of the output window and grinding the remaining surfaces using 3M "Imperial" polymer film.

Modification of the method of surface processing of highly hygroscopic Eu-doped scintillators [23] allowed to improve their light output for more than 10 % and resolution for 12.9–14.4 % as compared to specimens after the first processing step by grinding all their surfaces. Moreover, the modified method of surface treatment of specimens provides an improvement in their resolution by more than 11.1–11.4 % compared to the technical solution [23].

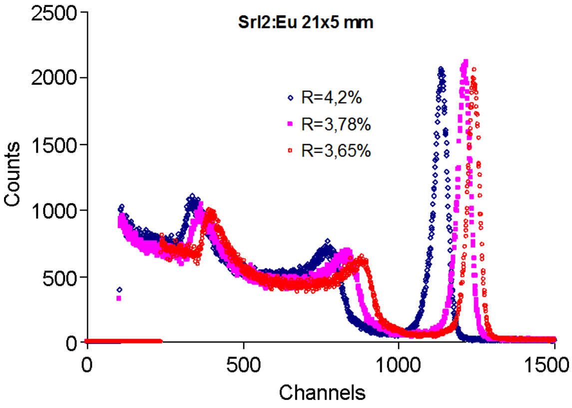

Fig. 4. Changes in the spectra of pulse amplitudes at different stages of surface processing of the SrI_2:Eu scintillator with a size of ∅21×5 mm (specimen No. 3).

The proposed modified method of processing the surfaces of scintillators with a diffuse reflector Tetratex TX3104 PTFE improves the resolution of scintillators with dimensions of ∅21×5 mm at 662 keV γ-ray energy up to 3.65 %.

Table 2 shows the sequence of operations for treating the surfaces of SrI_2:Eu scintillators with dimensions of 21×10 mm and the results of measurements of the energy resolution at various stages of their processing.

Table 2. Scintillation characteristics of ∅21×10 mm SrI_2:Eu detectors

Specimen No.	Processing stage	Sequence of operations	Resolution, %
5	1	1. Grinding of all surfaces with F 400 powder with an abrasive grain size of 20 microns in the composition with HMDS. 2. Flushing the polished surface of the HMDS	4.59
6	–	–	4.55
7	–	–	4.50
	2	For all samples: 3. Grinding with successive reduction in particle size from 14 to 6 μm. 4. Flushing the polished surface of the HMDS. 5. Sequential polishing of the scintillator surface from the side of the output window with LiF powder with a size of 5 μm and 3 μm and then washing with HMDS. 9. Processing of the side and entrance surfaces with 3M TM Imperial TM polymer film with the grain size of the main fraction:	
5	–	20 μm	3.98
6	–	25 μm	3.99
7	–	30 μm	3.97
5	3	Dry polishing of the scintillator surface from the side of the output window with 0.6 μm LiF powder	3.89
6	–	–	3.87
7	–	Dry polishing of the scintillator surface on the output window side with 0.3 μm LiF powder	3.87
4		Surface treatment in accordance with the technical solution [24]	4.37

Fig. 5 shows the change in the pulse spectra at different stages of the surfaces processing of the SrI_2:Eu scintillator No. 3 sized ∅21×10 mm.

From the analysis of the results given in Table 2 and Fig. 5 it follows that at the second stage of processing the surface of the specimens when changing from the polished surface of the specimens from the side of the output window to the polished one and grinding the remaining surfaces using 3M TM Imperial TM polymer film, there is an improvement in their light output and resolution. Additional dry polishing of output surface of the specimens using a powder with a grain size of abrasive of 0.3–0.6 μm provides an improvement of their light output to 10 % and the resolution to 10.9–11.5 %.

Modification of the method of surface treatment of specimens provides improved in light output of more than 10% and resolution of 12.9–14.4 compared with the specimens after the first processing step by grinding all of their surfaces. At the same time modification of the method of scintillator surface processing [23] provides 10.9–11.5 % improvement in resolution of the specimen with size ∅21×10 mm.

The modified method of surface treatment of scintillators with diffuse reflector Tetratex TX3104 PTFE improves the resolution of scintillators with dimensions of ∅21×10 mm at 662 keV gamma-ray energy to 3.87 %.

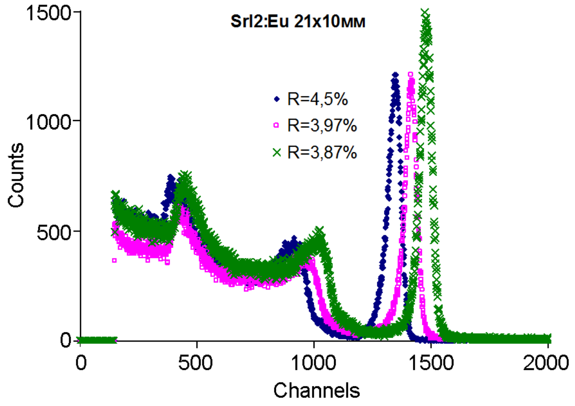

Fig. 5. Changes in the spectra of pulse amplitudes at different stages of surface processing of the SrI_2:Eu scintillator ∅21×10 mm (specimen No. 6).

Improvement of scintillation characteristics of a scintillators on the base of SrI_2:Eu single crystals with dimensions of ∅21×5 mm and ∅21×10 mm and high self-absorption can be explained as follows. Polishing of surface output window with consecutive decreasing of grain size to 0.3–0.63 μm leads to an improvement of optical characteristics of crystal by reducing the surface absorption. All this leads to an improvement in scintillation characteristics. The use of 3M TM Imperial TM film material with an abrasive grain size of 25–30 μm for grinding the inlet and side surfaces of the scintillator provides improved quality of the polished surface. The introduction of abrasive powder into the polymer matrix prevents them from falling out or being pressed into the surface of the crystal being processed, during processing and leaving large scratches on the surface. This leads to less disturbance of the crystal structure of the surface layer and high uniformity of the polished surface.

6. Conclusions

Options of surface treatment conditions of scintillators based on SrI_2:Eu crystal are selected using the computer simulation and experimental studies. All experimental results of study of influence of processing conditions on scintillation characteristics are in good agreement with the results obtained by computer simulation.

Obtaining the required degree of roughness of the inlet and side surfaces of SrI_2:Eu crystals using 3M TM Imperial TM film material and polishing their inlet surface with a successive decrease in grain size to 0.3–0.6 μm together with a Tetratex TX3104 PTFE diffuse reflector ensure improvement of resolution of scintillators with dimensions of ∅21×5 mm and ∅21×10 mm to 3.65 % and 3.87 % respectively. The implementation of these scintillation parameters is possible if liquids capable of absorbing residual moisture such as HMDS are used in the processing process.

Improving of the scintillators resolution will improve the quality of identification of radionuclides in the studied objects.

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