

Properties and Peculiar Features of Application of Isoelectronically Doped A^2B^6 Compound-Based Scintillators

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Abstract - The authors submit the data concerning the methods of obtaining semiconductor scintillators on the basis of the zinc chalcogenide crystal doped with impurities (Te, Cd, O, Me^{III} -metals Al, In, etc.). Characteristics of such crystals and mechanisms for the semiconductor scintillator luminescence are described as well. The scintillator luminescence spectra maximums are located within the range 450-640nm, which depends on the method of preparing the scintillator. The luminescence decay time ranges within 0.5-10 μ s and 30-150 μ s. The afterglow level is less than 0.01% after 10-20 μ s, and the radiation stability is $\geq 5 \cdot 10^8$ rad. Thermostability of the output characteristics of new semiconductor scintillators on the basis of zinc selenide is prescribed by thermodynamic stability of the principal associative radiative recombination centers that come into existence due to the crystal lattice inherent imperfections. Certain application fields of the new scintillators are examined taking into account their particular qualities.

Key words : A^2B^6 compounds, semiconductor scintillator, luminescence properties, radiation detectors

INTRODUCTION

For achieving the progress in radiation instrument-making industry (such as X-ray medical and technical tomography - as well as X-ray introscopy in general), dosimetry of high-power flows of ionizing radiation, etc., one requires scintillating materials characterized by a series of corresponding optimal properties. They are the following: a high rate of response, almost total absence of afterglow after 10-20 μ s, thermal- and radiation stability of the output parameters. Besides, the scintillator emission spectrum must be shifted towards the red region, which improves the spectral matching with up-to-date photo detectors - e.g., photodiodes. The scintillating materials must not be hygroscopic, etc. However, some of the best scintillators (such as NaI(Tl), CsI(Tl)) are characterized by a low radiation stability,

which, in fact, does not exceed 10^4 rad, and a high afterglow (up to several percent after 20s). In addition, these scintillators are hydrolabile (hygroscopic), which causes additional difficulties under the conditions of high humidity. Low conversion efficiency is inherent in oxide crystals such as CdWO₄ (CWO), PbWO₄ (PWO), Bi₄Ge₃O₁₂ (BGO), Gd₂SiO₅ (GSO) [1]. Therefore, obtaining of new highly efficient scintillators, study of their properties and elaboration of the corresponding techniques are surely of great importance for many fields, the above-mentioned ones included, where such materials are applicable.

The use of A^2B^6 -type crystals with isovalent dopants (IVD) is one of approaches prospective for solving this problem. After the first works by Watkins, Tomas, Fistule et. al, it has been found out [2-5] that IVD alters the process of imperfection formation in the matrix lattice and

stimulates formation of stable associations of inherent point imperfections (PI) with IVD, which determine the crystal optical characteristics. Further, in [6] the following is reported. First, interaction of PI with IVD is explicable by the existence of short-range forces acting between them (30-100Å), which bind PI and IVD together into complexes. Second, there exists a long-range resilient potential that favors the PI formation. If IVD dimensions exceed sizes of the matrix atom that substitutes for IVD, the vacancy concentration increases. If IVD is introduced in small quantities (about 1% of all atoms), only the neighboring atoms interact. That is, IVD changes the vacancy concentration just in the neighboring sublattice. And what is more, the IVD introduction favors a decrease in the concentration of PI complexes in comparison with other admixtures. This can cause the impurity extraction from the crystal if the condition is satisfied [7]:

$$C_{IVD}/C \approx (N_{IVD}/N) \cdot \exp[(Q_1 - Q_2)/kT] > 1,$$

here N_{IVD} and N denote the concentration of IVD and that of another complex-forming impurity, respectively; correspondingly, Q_1 and Q_2 designate the energy binding the complex imperfection with IVD and with another impurity.

THE ANALYTICAL GROUNDING

Aiming at elaborating a new scintillating material, we have postulated that it is possible to make highly efficient scintillators with predictable emission spectra on the basis of the A^2B^6 -type crystals if IVD and the atom to be replaced substantially differ in the ion radiuses and electronegativity. This statement is based on the fact that in crystals containing IVD the radiation center parameters are prescribed by the crystal lattice PI type that prevails [8-12]. As a crystal matrix, we have chosen zinc selenide, while tellurium and

oxygen serve as the basic IVD.

Making use of the equation of electroneutrality and the effective charge concept (e^*), one can estimate the influence of Te as IVD on formation of defects in the ZnSe lattice [10]:

$$[V_{Zn}]e^{*ZnSe} - [Zn_i]e^{*ZnSe} - [V_{Se}]e^{*ZnSe} + [Se_i]e^{*ZnSe} - (e^{*ZnSe} - e^{*ZnTe})[Te_{Se}] = 0,$$

here $[V_{Zn}]$, $[Zn_i]$, $[V_{Se}]$, $[Se_i]$ denote the concentrations of vacancies and interstitial atoms in sublattices of Zn and Se; $[Te_{Se}]$ is the concentration of Te, used as IVD and substituting for the atom of Se. Besides, e^{*ZnSe} is the effective charge of Zn-Se compound; e^{*ZnTe} is the effective charge of Zn-Te compound; $e^* = [N\lambda^2 - (8-N)] / (1 + \lambda^2)$, where $1 - \lambda = 1 - \exp[-0.25 \times (X_x - X_{Zn})]$ [13]; N is valency; λ is covalence degree; X is electronegativity ($X_{Zn}=1.5$; $X_{Se}=2.4$; $X_{Te}=2.1$). The substitution yields $e^{*ZnSe}/e^{*ZnTe} \approx 0.918$. Taking into account that $[Zn_i] < [V_{Zn}]$ (the interstitial zinc mobility substantially exceeds the zinc vacancy mobility) [10] and under the supposition that $[V_{Se}] \approx [Se_i]$, one finally gets:

$$[V_{Zn}] \approx (1 - e^{*ZnTe}/e^{*ZnSe})[Te_{Se}].$$

That is, the zinc vacancy concentration increases in direct proportion to the concentration of Te IVD. Further, by the analytical and experimental methods, it is found out that the introduction of Te isovalent admixture from outside enhances formation of defects in the Zn sublattice and favors the augmentation of V_{Zn} concentration. The addition of Te as IVD stimulates the formation of stable not mobile associations $V_{Zn} + Te_{Se}$ and mobile interstitial zinc, which make the main elements of the radiative recombination centers and are responsible for the principal working band of the scintillator luminescence ZnSe(Te) - 630-640nm. The semiconductive scintillator of the given type (SCS-I, or the "classical" one) is already widely applied in some areas of the radiation instrument-making industry [1,11].

If the condition

$$[O]/[Te] < [\exp(-0.25 \Delta X_{ZnSe}^2) - \exp(-0.25 \Delta X_{ZnTe}^2)] / [\exp(-0.25 \Delta X_{ZnO}^2) - \exp(-0.25 \Delta X_{ZnSe}^2)]$$

is justified, it serves as an additional factor that influences the formation of luminescence centers responsible for the radiation band $\lambda_{max}=630-640\text{nm}$ in SCS-I crystals [10, 14]. Here ΔX_{AB} is the difference between the electronegativeness of the metal A (Zn) and chalcogenide B (Se, Te or O). Introduction of oxygen in active forms (O^0 , O^- , etc.) - as well as oxygen and metals of group III (Al, In, Ga, etc.) - into ZnSe crystals in the amount about 1/5 part of the Te concentration alters the luminescence center dominating type. The new semiconductor scintillators, recently made by this technique (SCS-II or "quick" scintillators), essentially differ from SCS-I in their luminescent characteristics. They are prospective for making fast-acting introscopy systems and other devices of the radiation control [14, 15]. Below we will briefly dwell on the methods of obtaining SCS on the basis of doped zinc selenide and sulfide and describe their properties.

EXPERIMENTAL

One of the mostly widespread ways of obtaining crystals on the basis of binary and multicomponent compounds of A^2B^6 -type is the method of crystal growing from the melt under pressure of an inert gas - Bridgeman-Stockbarger's method. It permits obtaining SCS crystals of the diameter up to 40 mm and the weight about 1 kg in a relatively short time (several days). The inert gas high pressure (up to 10^7 Pa) permits reducing the starting substance mass transfer from the crystallization zone to the cold area. This process is realizable due to the fact that the compound components dissociating in the gaseous phase are subjected to high pressures. During the process of SCS crystal growth, the temperature is being

heightened up to the value that by 50-100°C exceeds that of the compound melting. After this, the crucible containing the starting furnace charge is being uprightly transported through this zone at the speed 2.5-25mm/hour. After the passage through the maximum temperature area, the melt is crystallized in the temperature gradient zone 30-100degrees/cm. Being the most efficient, the given method implies the use of relatively simple equipment. Besides, the method provides a sufficient reproducibility of parameters of the scintillators on the basis of A^2B^6 doped with IVD and (or) Me^I (Cu, Ag, etc.), Me^{III} (Al, In, Ga, etc.). Because of high temperatures and corrosive media where crystals are grown, only graphite can be used as a material for the crucible, being relatively resistant to these factors. Special demands for the graphite purity, the composition of admixtures and the starting raw material stoichiometry are stipulated in the first place, they concern the presence of admixture components in the raw material intended for the SCS crystal growing. For making solid solutions of $ZnS-Me^{II}X$ (here $Me^{II}X$ denotes zinc, cadmium or mercury chalcogenide in the concentration 3-10% of the total weight), the starting mixture is subjected to the preliminary agglomeration and alloyage in the growing vessel. The raw material prepared in this way can be used for making crystal boules. Further the latter are used for forming the SCS-I type scintillating material by their annealing in Zn vapor. In making SCS-II scintillators, an intermediate stage is introduced. At this stage, ZnSe is activated with oxygen-containing admixtures [14]. Besides, in order to improve solvability of oxygen and its fixation in the lattice-matrix ZnS or ZnSe, metals of the group I (Me^I) or group III (Me^{III}) can be supplementary introduced - either in the elementary state or in the chalcogenide form.

The crystal aftergrowth annealing in Zn (or Cd) vapor (1000-1300°C, 24-72hours) also makes an important stage in the process of making SCS crystals of the I- and II types. At this stage, the formation of luminescence

centers is finished. In addition, one also suppresses possible channels of non-radiating recombination and levels that condition the scintillator persistence.

RESULT AND DISCUSSION

SCS luminescent characteristics

X-ray luminescence (XRL) spectra of crystals SCS-I and SCS-II are given in Fig.1. As regards SCS-I crystals, in the temperature range 80-400⁰K exists just one band of the radiation emission $\lambda_{\max}=635\text{nm}$. Under the temperature 300⁰K, the maximum of the radiation emitted by crystals SCS-II ranges within 600-620nm. As the temperature decreases, the maximum is initially shifted to the band 630-640nm. When $T<230^0\text{K}$, the peak returns into the starting position. For crystals SCS-I, the principal working band ($L=635\text{nm}$) has two slopes in the dependence $\ln I_0=f(10^3/T)$. As a rule, this is typical of the donor-acceptor recombination. However, in this band - as well as in the band $L=605\text{nm}$ the maximum position is not being displaced as the XRL excitation level is varying. There are no temporal changes in forms of spectra of the bands $L=635\text{nm}$ and $L=605\text{nm}$, which excludes the possibility of their being of the donor-acceptor nature. As the two slopes in the dependence $\ln I_0=f(10^3/T)$ can indicate that an intricate associative acceptor participates in the process [18-23], the appearance of the bands

$L=635\text{nm}$ and $L=605\text{nm}$ can be interpreted as manifestation of radiative transitions of the "conductive zone - associative acceptor" type. Centers of the types $(V_{\text{Zn}}\text{Te}^{\circ}\text{Se})^-$, $(V_{\text{Zn}}\text{Te}^{\circ}\text{SeO}^{\circ}\text{Se})^-$ or other can play the role of a complex acceptor [1, 9-12, 14]. The analogous centers with the participation of V_{Zn} , Te, Os, etc. are responsible for the luminescence in SCS of the type ZnS(IVD). Under low temperatures and high levels of SCS crystal interband excitation with the help of a laser - as well as in cathodoluminescence spectra (CS) - a series of bands of the near-edge radiation emitted by excitons in the range 440-500nm is observable. The intensities of the bands $E_D(A_1)$ and $E_D(A_2)$ correlate, respectively, with the concentrations of oxygen and tellurium in SCS crystals. Under 4.2⁰K, in XRL and photoluminescence (PL) spectra of SCS crystals of both types, there also appears a weak band of the acceptor-donor nature ($\lambda_{\max}=950-980\text{nm}$). Probably, it is induced by the presence of zinc isolated vacancies V_{Zn} . The decay time τ of SCS crystals substantially depends both on the type of exciting radiation and on its power. In the cases of excitation with X-rays ($U_a=40-160\text{keV}$, $i_a=0.1-5\text{mA}$, $\tau_x>1\mu\text{s}$) and PL, for SCS-I $\tau_x=50-150\mu\text{s}$ and for SCS-II $\tau_x=1-5\mu\text{s}$. In the case of SCS irradiation with electron pulses ($E_e=300\text{keV}$, $j_e=0.5\text{A/cm}$), $\tau_x=10-30\text{ns}$ [1, 11]. In INP of NAS of Uzbekistan kinetics of highlighting of crystals SCS-I and SCS-II has been measured in the counting mode with making use of γ -radiation emitted by the isotope ²⁴¹Am. The measurements have demonstrated that in the case of the given scintillators the scintillating flash duration, being shorter than that of the "one-electron" pulses coming from a fast-response photo electron device (PED), belongs to the subnanosecond range. In this respect, it is not inferior to one of the quickest inorganic scintillators NE 843, based on ZnO(Ga) [20]. Under the same conditions of measurements but when sources of γ -radiation of higher power are used (¹³⁷Cs, etc.), the scintillating flash duration increases more than by the order of

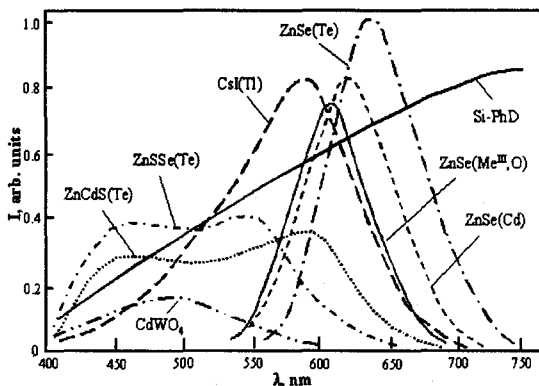


Fig. 1. Spectral features of radiation of A^2B^6 scintillators.

magnitude. Measurements of the X-ray luminescence kinetics indicate that after 20s the SCS-I crystal persistence level does not exceed 0.05%. In the case of the SCS-II crystals, this parameter is lower than 0.01% already after 10 μ s.

Radiation stability of SCS

In the given work, the term "radiation stability" of scintillators (RS) implies stability of SCS output characteristics (the light output, the afterglow level, transparency, etc.), measured under the effect of ionizing radiation of various types and in various doses. SCS crystal RS is investigated under the condition of the irradiation with γ -rays ($E_{\gamma} \approx 1$ MeV, $D_{\gamma} \leq 10^9$ rad), protons ($F_n \leq 10^{19} \text{ cm}^{-2}$), electrons ($E_e = 0.54\text{--}2.26$ MeV, $P_e = 3 \cdot 10^9 \text{ cm}^{-2} \text{ c}^{-1}$, $D_{ee} \leq 5 \cdot 10^7$ rad) and reactor neutrons ($F_n \leq 10^{19} \text{ cm}^{-2}$) [24]. The SCS crystal irradiation with γ -rays ($D_{\gamma} \leq 5 \cdot 10^8$ rad) diminishes the XRL by 15–20%, the spectral-kinetic characteristics remaining unchanged. The electron doses smaller than the equivalent ones ($D_{ee} = 5 \cdot 10^7$ rad) do not affect the SCS XRL parameters. When $F_p = 8.7 \cdot 10^{14}\text{--}1.7 \cdot 10^{15} \text{ cm}^{-2}$, processing of the crystals with protons causes degradation the SCS crystal optical characteristics: the light output decreases by 90%, transmission is deteriorated both in the visible and IR spectrum regions. When SCS crystals are irradiated with neutrons, the light output increases by 10–150%, whereas the XRL spectrum-kinetic characteristics remain unchanged. Besides, an increase in the differential absorption is detected in the range 480–550nm, simultaneously accompanied by a decrease in the sample specific resistance i.e., one can observe the changes characteristic of ZnSe(IVD) crystals after their annealing in Zn vapor. Any additional manifestations of the physical-chemical processes, observable in other compounds of A^2B^6 -type, are not registered [25] in SCS crystals, which can also testify their high RS. The RS high value and the thermal stability of SCS crystal output parameters are conditioned by the generation of thermodynamically

stable and non-recombining associations ($Zn_i\text{--}V_{Zn}\text{--}X_{Se}$; here X is a chalcogenide Te, O, S in the compound with Me^{III}). Besides, an optimal combination of elastic interaction of PI and IVD with Coulombian interaction during the processes of excited state relaxation also plays a part in heightening of the RS value and achieving the thermal stability of the above-mentioned parameters [1, 8, 24].

The mechanism for SCS luminescence

Building the model of radiative transitions in SCS crystals, one should start from the following facts, established experimentally and analytically [1, 2, 8, 9–12, 14]:

1. It is unnecessary to introduce any other foreign admixtures into the luminescent centers in addition to the inherent PI or zinc selenide lattice components, defect complexes on their basis and tellurium- or oxygen IVD.

2. The principal working bands of SCS-I and SCS-II ($L=635$ nm and $L=605$ nm) are induced by the recombination mechanism for luminescence.

3. Zn fine donors remain in the ionized state down to the temperatures of liquid helium ($E_a = 11\text{--}20$ meV), which provides the electron high density ($n_e \sim 10^{18} \text{ cm}^{-3}$) in the crystal conductivity zone. That is, in atoms of Se, $4p^4$ -shells are completely filled and $4p^5$ -shells are filled partially.

4. The center of radiative recombination of the bands $L=635$ nm and $L=605$ nm is associative [1,8,11].

The thermodynamic analysis given to the processes of defect formation in SCS indicates that if IVD concentrations are small, the concentrations of zinc additional vacancies are proportional to them $-[V_{Zn}] \sim [Te_{Se}]$, and the energy of migration of zinc atoms Zn_i (and their evaporation) decreases [2,10]. Introduction of Te IVD is accompanied by a steep increase in the SCS luminescence intensity. Consequently, together with defects V_{Zn} , tellurium forms associative centers of radiative recombination of the type $(Te_{Se}^0 V_{Zn})^-$ [1, 8, 9, 11]. For crystals SCS-II (the principal working band $L=605$ nm) formation of associations of the

types $((O_{Se}^{\circ}Te^{\circ}V_{Zn}^{-}), (O_{Se}^{\circ}V_{Zn}^{-})$ or $(O_{Se}V_{Zn}Me^{\text{III}}_{Zn}Zn_i)$ is possible [12, 14]. The excited charge carrier capture cross-section even exceeds that of the centers $(Te^{\circ}SeV_{Zn}^{-})$. In the scheme of radiative transitions given in Fig.2, the pattern of formation and behavior of optically active centers in SCS are taken into

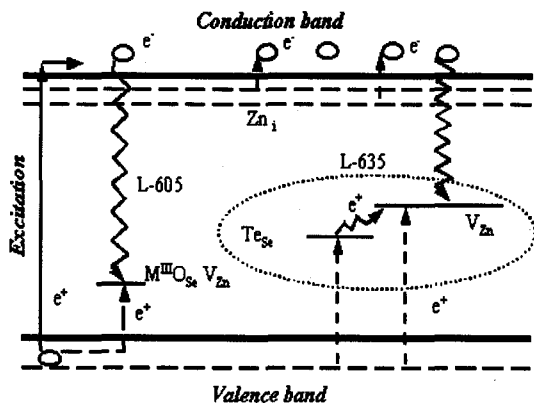


Fig. 2. Scheme of non-radiative and radiative transitions in ZnSe(IVD) crystals.

account. If in SCS crystals exist luminescence centers of both kinds (one characterized by a higher activation energy and another of the capture cross-section of a larger size), the spectral composition of the radiation emitted by SCS has to be changed with variations in the sample temperature. This phenomenon has been observed in the tests on the XRL thermal extinguishing of such crystals [14, 15]. The radiative transitions in SCS of the ZnS (IVD)-type where participate the centers $(V_{Zn}, Te_s, O_{Se}, \text{etc.})$ occur according to the similar schemes.

SPECIFIC FEATURES OF SCS APPLICATION

In Table 1, the summary properties of new SCS are presented. They give a concept of the crystal optical, scintillating and performance

Table 1. Comparison between best traditional scintillation and new A^2B^6 -based SCS.

Parameter	Scintillators				
	<i>ZnSe(Me^{III},O)</i>	<i>ZnSe(Cd,Hg)</i>	<i>ZnSe(Te)</i>	<i>CsI(Tl)</i>	<i>CWO</i>
Density ρ , g/cm ³	5.42	5.42	5.42	4.51	7.99
Effective atomic number, <i>Z</i>	33	33	33	54	65
Hygroscopicity	no	no	no	yes	no
Luminescence maximum λ_{max} , nm	590-610	615-625	630-640	550	490
Afterglow, σ : after 5 ms, % after 20 ms, %	< 0.01 ≤ 0.005	< 0.02 < 0.01	< 0.2 < 0.05		< 0.05 ≤ 0.02
Absorption coefficient of intrinsic luminescence, cm ⁻¹	0.1-0.2	0.1-0.3	0.05-0.3	< 0.05	< 0.05
Light output with respect to CsI(Tl) for 2 mm thickness, % (in combination with photodiode)	90-110	100-120	100-140	100	30
Decay time, τ , μ s	0.5-10	5-20	50-150	1	10-15
Coefficient of spectral matching with PD, Ku	0.9	0.91	0.92	0.75	-
Light yield, photons/MeV	up to $5 \cdot 10^4$	up to $4 \cdot 10^4$	$7.5 \cdot 10^4$	$5.5 \cdot 10^4$	$1.3 \cdot 10^4$
Radiation stability, rad	$1 \cdot 10^8$	$3 \cdot 10^8$	$5 \cdot 10^8$	$5 \cdot 10^4$	$1 \cdot 10^8$

characteristics. Besides the evident advantages (such as light output, inertia, radiative and climatic (exploitation) stability), SCS have a substantial drawback - i.e., a relatively high coefficient of the optical absorption $\alpha(\lambda)$ in the area of the radiation spectrum maximum. In addition, their effective atomic number Z_{ef} is relatively low (for ZnSe $Z_{ef}=33$ and for ZnS $Z_{ef}=27$). Both parameters (α and Z_{ef}) put certain limitations on the optical and radiative lengths of the scintillating elements. In this connection, multi-energy X-ray introscopy and tomography are the best areas of applying the new SCS [1, 15]. There the latter are used in the low-energy subsystem of the detecting section (the scintillator thickness makes 0.5-5mm). As SCS is a unique substance, which combines the properties of semiconductor and scintillator, one is interested in making scintillating elements that go with a photodetector of the type ZnSe(Te)-(ZnTe-CdSe) [16], introduced in the form of a superficial heterostructure. If a Schottky structure of the type ZnSe(Te)-Ni is to be formed on the SCS crystal surface, such element is applicable as an efficient sensor of UV radiation of the ranges A, B and C [17]. As the parameters α/r , β/r substantially differ from unity in the case of SCS of the types ZnSe(Cd) and ZnS(IVD), these scintillators, combined with other ones (such as CsI(Tl), CWO, polymer scintillators, etc.) are applicable in joint detectors, intended for the separate detection of ionizing radiation of different types that belong to mixed radiation flows. In addition, taking into account extremely high values of the SCS radiation stability and light output, one can see that the given material is prospective for the use in dosimetry of high-power flows of ionizing radiation.

CONCLUSIONS

As regards the XRL light output, SCS crystals are not inferior to the best alkali halide crystal (AHC) scintillators, and they are several

times better than oxide scintillators of the types BGO, GSO, PWO and CWO. As for the radiation stability, SCS crystals are comparable with oxide scintillators, this parameter being more than 10^3 times as high as that of AHC. The luminescence kinetics, physical-chemical characteristics and electric parameters of SCS crystals are conditioned by particularities of the capture center formation and radiative recombination in the semiconductors characterized by a wide forbidden band. These particular features make SCS applicable in sensors of various kinds not only in X-ray introscopy systems, but in other branches of radiative control and nuclear investigations.

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