# Scintillation crystals based on solid solutions of oxyorthosilicates

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Structure, mechanical, optical and scintillation properties of oxide scintillators based on solid solutions of rare-earth oxyorthosilicates are considered. Characteristics of the developed cerium doped lutetium-gadolinium silicate  $\text{Lu}_{2-2x}\text{Gd}_{2x}\text{SiO}_5$  crystals are described versus best world analogues. Afterglow level decrease by several times and drop of slow decay component contribution are the crucial features of the obtained crystals. Further prospects of work dealing with optimization of LGSO:Ce scintillation properties are discussed.

Рассмотрены структура, механические, оптические и сцинтилляционные свойства оксидных сцинтилляторов на основе твердых растворов оксиортосиликатов редкоземельных элементов. Характеристики разработанных кристаллов лютеций-гадолиниевого силиката  $Lu_{2-2x}Gd_{2x}SiO_5$ , активированного церием, сопоставляются с лучшими мировыми аналогами. Отличительными свойствами полученных кристаллов является снижение уровня послесвечения в несколько раз и уменьшение вклада медленного компонента затухания сцинтилляций. Обсуждаются перспективы дальнейших работ по оптимизации характеристик данного класса сцинтилляторов.

#### 1. Introduction

Oxide single crystals based on rare-earth silicates are known scintillators used in devices of medical diagnostics, scientific experiments on high-energy physics and particle physics, other applications [1]. Light yield up to 30000 phot/MeV and more, nanosecond decay, high density (~7 g/cm<sup>3</sup>), radiation and chemical stability, and nonhygroscopicity are their basic advantages [1, 2]. At present, only Ce-doped oxyorthosilicates are found vast practical application due to high scintillation efficiency of this activator in the given hosts [3]. Luminescence of other fast dopants, in particular Pr<sup>3+</sup>, is quenched at room temperature. Many publications are devoted to study of this phenomenon, for instance [4-6].

Two major tendencies of works on development of Ce-doped oxyorthosilicates can be emphasized. The first case deals with codoping

with small concentration (up to 1-2 at.%) of another element without significant influence of the host structure [7, 8]. In most cases codoping not leads to substantial improvement of scintillation or other characteristics. The most successive examples providing increase of light yield is codoping with divalent cations, especially Ca<sup>2+</sup> [2, 9]. Ca<sup>2+</sup> ion is isomorphic to Ce<sup>3+</sup> due to coincidence of ionic radii (1.00 and 1.02 Å, correspondingly). Its positive influence on scintillation characteristics is connected with possibility to decrease quantity of oxygen vacancies by addition of divalent cation [9]. Improvement of crystal growth stability due to surface tension increase of melt meniscus by Zn<sup>2+</sup> doping was also noted [2]. Ce-Yb codoping provides decrease of light storage in LSO [10, 11]. LSO:Ce,Yb crystals with improved radiation hardness and decreased afterglow level by the factor of 100 in comparison with  $Lu_2SiO_5$ :Ce (LSO:Ce) were reported in [11]. The work on correction of crystal stoichiometry by addition to melt of 1.9 at.% excess of  $\text{Li}_2\text{O}$ ,  $\text{Lu}_2\text{O}_3$ , or other oxide of trivalent rare earth cation also can be ascribed to this group [12]. As the result, improvement of light yield by 5–38 % and decrease of decay time by 1–7 ns is achieved. In accordance with [12] this codoping provides filling of vacancies formed in crystal lattice owing to partial situation of  $\text{Lu}^{3+}$  ions in interstitial positions.

The second approach to modify scintillation characteristics of oxyorthosilicates is obtaining of crystals based on  $(RE1)_{2x}(RE2)_{2-2x}SiO_5$  solid solutions (RE1 = Gd, Lu; RE2 = Y, Lu). It will be considered in more detail in the present paper.

### 2. Growth and structure of single crystals

 $Gd_{2x}Y_{2-2x}SiO_5$ : Ce (GYSO:Ce) and

 $Lu_{2x}Y_{2-2x}SiO_5$ : Ce (LYSO:Ce). Oxyorthosilicates demonstrate congruent melting for all the range of lanthanides from La3+ to Lu3+, for this reason they can be easily crystallized from melt [13]. Silicates with bigger ionic radii (Ce-Dy) and smaller ionic radii (Ho-Lu) possess monoclinic space symmetries  $P2_1/c$  and C2/c, respectively. Crystals with symmetry of the first type are intended to cracking; crystals of the second type demonstrate better plasticity [14].

Substitution of rare earth ions in solid solution crystals leads to change in lattice parameters and, in some cases, in space symmetry type. Control of host structure and composition enables improvement of scintillation characteristics and mechanical properties of crystals, as well as lowering of crystallization temperature and production cost. However, only materials containing optically inactive cations Gd<sup>3+</sup>, Lu<sup>3+</sup>, and Y<sup>3+</sup> at Ce<sup>3+</sup> doping showed themselves as efficient scintillators [15–22].

Improvement of mechanical characteristics at doping of GSO:Ce by Y3+ is a successful example of the second approach. Decrease of elementary cell volume by 0.6 % at addition of 20 % Y3+ evidences the effect of volume compensation in crystal [19]. As the result, plasticity of crystals improves, and cleavage by the (100) plane weakens. At the same time, no notable change of scintillation characteristics is observed [19, 20].

Inhomogeneity of activator distribution in boules is well-known factor leading to crystal quality degradation [22]. Doping of LSO with yttrium improves uniformity of characteristics in volume due to increase of Ce<sup>3+</sup> segregation coefficient [23]. LYSO crystals are not behind LSO by scintillation characteristics. LYSO, together with LSO and Gd<sub>2</sub>SiO<sub>5</sub> (GSO), are widely applied because of lower by 100°C crystallization temperature and better stability of growth process [21, 23, 24].

In common, mixed crystals with P2<sub>1</sub>/c structure (GYSO) are characterized by moderate light yield around 10000 phot/MeV and low afterglow level. The observed light yield is linked to temperature quenching of sevenfold coordinated Ce<sup>3+</sup> luminescence [25]. Crystals with C2/c structure based on LSO and LYSO demonstrate much higher light yield (25000–30000 phot/MeV), high afterglow level (around 0.5 % after 3 ms). At present, LSO and LYSO with diameters up to 100 mm and length up to 250 mm are produced in industrial scale (see for ex., [21]).

 $Gd_{2x}Lu_{2-2x}SiO_5$ : Ce (GGSO : Ce). Structure of LGSO solid solution crystals can correspond to the both abovementioned symmetry types [17]. There was no systematic study of structure and scintillation properties of LGSO depending on Lu/Gd ratio, except [17]. No information about industrial scale production of these crystals was found. LGSO compared to other orthosilicate solid solutions demonstrates big difference in ionic radii of host cations (0.86 Å for Lu<sup>3+</sup> and 0.94 Å for Gd<sup>3+</sup>). In addition, structure type of LGSO crystals depends on Lu/Gd ratio. These factors complicate the obtaining procedure of single crystals. At the same time, LGSO was found to demonstrate the complex of properties not observed in other known orthosilicates.

To study structure and scintillation properties of LGSO, full range of solid solution crystals from GSO to LSO was grown by the Czochralski method. Crystals were grown from Ir crucibles with the dimensions  $60{ imes}60$  mm. The pulling rate was 1.5-3 mm/h, the rotation rate was 25-35 rot/min. The technology described in [26] and based on utilization of upper heater was applied to minimize crystal cracking. Post-growth annealing in low-gradient temperature field in inert atmosphere at 1500°C was conducted to eliminate thermal stresses in crystals. The diameter of grown crystals was 30-35 mm and the length was 30-60 mm (Fig. 1). Some crystals contain cracks owing to discrepancy of lattice parameters of the seed and grown







Fig. 1. Photos of as-grown LGSO:Ce crystals.

crystals and not completely optimized thermal conditions. Majority of crystals contain substantial quantity of macroscopic inclusions; composition of inclusions has not been identified yet.

It was shown that polymorph transition between structure modifications is observed at Lu concentration around 20 at. % [27]. The interesting features are that LSO-type structure persists even at substitution of 80 % Lu by Gd, and crystals of the both structure modifications can be obtained in the concentration range 10-20 % Lu depending on growth conditions. It is known that trivalent cations in oxyorthosilicates occupy two unequivalent positions: 9- and 7-coordinated sited in the GSO-type structure and 7- and 6-coordinated sited in LSOtype structure [13]. Herein, in the LSO-type structure the average bond length between trivalent cation and surrounding oxygens is by 0.1 Å larger compared to LuO<sub>6</sub> (Fig. 2). This difference is the cause of non-uniform distribution both activator and host cations in LGSO:Ce. It was shown experimentally that bigger ions of Gd<sup>3+</sup> tend to incorporate into 7-coordinated site. The distribution coefficient of Gd in LSO is 0.72 for 7-coordinated site and 0.54 for 6-coordinated site [28]. Luminescence spectra (they will be considered in more detail below) evidence that Ce<sup>3+</sup> ions with larger ionic radius are also prefer to occupy 7-coordinated positions. In addition, Lu/Gd ratio and activator concentration also influences Ce distribution between polyhedra. Redistribution of intensity from CeO<sub>6</sub> peak (475 nm) for CeO<sub>7</sub> peak (420 nm) at X-ray luminescence spectra correlates with increase of light

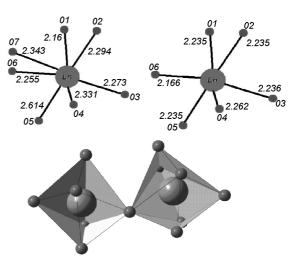


Fig. 2. Above - structure of  $SiO_4$  and  $LnO_6$ , LnO7 polyhedra in orthosilicates (monoclinic space symmetry C2/c); below - structure of  $Si_2O_7$  and  $LnO_7$  polyhedra in pyrosilicates (orthorhombic modification  $P_{mna}$  of GPS). One of non-silicon-bound oxygens in C2/c structure is denoted by the arrow.

yield of crystals by 20-30 % and improvement of energy resolution to 6.5-7 % under  $\gamma$ -rays (662 KeV) [29]. This patent also claims a method for obtaining crystals with improved characteristics.

Presence of silicon-non-bound oxygens forming bridges between lanthanide atoms is the another feature of oxyorthosilicate structure (see Fig. 2). In particular, strong afterglow (up to several percents) in LSO and LYSO [16] is attributed to vacancies formed at these oxygens. Likewise in LYSO, addition of Gd<sup>3+</sup> with bigger ionic radius into LSO host leads to increase of lattice

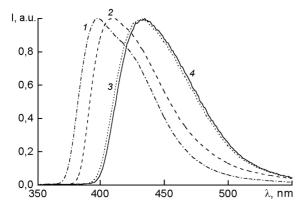


Fig. 3. X-ray luminescence spectra of Cedoped rare earth oxyorthosilicates: I — LSO, 2 — LGSO (55 % Lu), 3 — GSO, 4 — GYSO (15 % Y).

constants and increase of Ce<sup>3+</sup> segregation coefficient from 0.2 to 0.65 [27].

Mechanical properties of LGSO:Ce were studied in the paper [30]. Microhardness of crystals with  $P2_1/c$  structure is varied within the range 6-9 GPa. Transfer to C2/c (LSO-type) structure and further increase of Lu content leads to monotonous increase of microhardness from 10 to 19 GPa. It is a substantial factor at mechanical treatment of crystals.

### 3. Optical and scintillation characteristics

Light yield and energy resolution of scintillators under γ-irradiation were determined on elements with the dimensions  $10\times10\times2$  mm<sup>3</sup> by the method based on the measuring of intrinsic resolution of the photomultiplier. Decay curves under γ-radiation (137Cs, 662 KeV) were obtained by a single-photon technique. Parameters of thermally stimulated luminescence were determined at a heating rate of about 5 K/min. Afterglow kinetics after X-ray irradiation with the dose 5 R was measured using a special purpose measurement setup. X-ray luminescence was measured at excitation by X-ray tube with copper cathode (40 mkA, 40 kV). Radiation from the irradiated samples was registered by a FEU100 photomultiplier. Detailed description of the measurement procedures can be found in the works [19, 31-33].

Luminescence bands of oxyorthosilicates are situated in the range 410-550 nm (Fig. 3). Spectral composition of luminescence and scintillation characteristics of LGSO:Ce depend on cation ratio in the host and structure type [27, 28]. Light yield

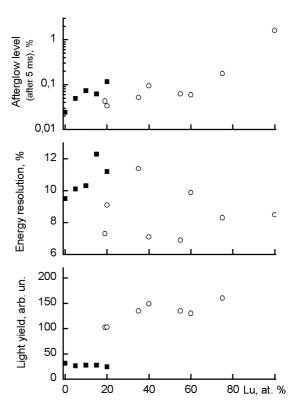


Fig. 4. Light yield under  $\gamma$ -radiation, energy resolution (662 KeV) and afterglow level (after 5 ms) in LGSO:Ce ( $10\times10\times2$  mm³) vs. host composition. Hollow and filled symbols correspond to crystals with monoclinic symmetries C2/c and P2<sub>1</sub>/c, correspondingly.

sharply increases near 20 % Lu at polymorph transition. Then, it smoothly increases with Lu content (Fig. 4). For instance, the measured absolute light yield of  $Lu_{0.8}Gd_{1.2}SiO_5$  crystal is 21300 phot/MeV [35].

Energy resolution of samples with C2/c structure is substantially higher. The best results in the range 6.9-7.3~% at  $(662~{\rm KeV})$  are observed at Lu concentration between 20 and 60 at. % (see Figs. 4, 5). About the same parameters (up to 6.5~%) were obtained in [36]; this is a record value for silicate scintillators. Deterioration of energy resolution in LSO:Ce [37] is attributed to energy transfer from CeO<sub>7</sub> to CeO<sub>6</sub> center. Logically to suggest that conditions of this negative process should get worse in LGSO at minimization of CeO<sub>6</sub> quantity and increase of distances between the centers.

Decrease of afterglow level by several times in LGSO in comparison with LSO and known LGSO analogs is an important result of our work. Afterglow sharply decreases at addition of 30 at. % Gd<sup>3+</sup> into the host and

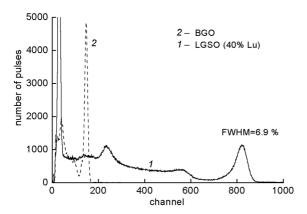


Fig. 5. Pulse height spectra of LGSO:Ce (1)  $(10\times10\times10 \text{ mm}^3)$  in comparison with BGO (2)  $(10\times10\times10 \text{ mm}^3)$  at  $\gamma$ -irradiation <sup>137</sup>Cs (662 KeV).

reaches its minimum at 40 % Gd (60 % Lu), see Fig. 4. According to [38], the reached afterglow level after 20 ms is called only by intrinsic radioactivity of <sup>176</sup>Lu isotope that can not be avoided in Lu-containing scintillators. Herein, peaks above room temperature associated with oxygen vacancies disappear in thermoluminescence glow spectra, as well as peak near 100 K linked to shallow traps are suppressed [27]. Similar data on TSL are presented in [39], however, there are no data presented on afterglow level in millisecond range, and no possibility to compare it with our results.

In [40], afterglow and high temperature TSL peaks in LSO and LYSO are attributed to process of thermally stimulated tunneling recombination between electrons localized on traps on oxygen vacancies and holes captured by activator ions. Intensity of this process is inversely proportional to exponent of trap-to-activator distance, therefore, even small change of the distance matters. Afterglow in LGSO:Ce at large Gd

concentrations can be suppressed by this mechanism, because the distance between vacancies on oxygen atoms and activator ions increases by 0.05-0.1 Å due to substitution of Lu<sup>3+</sup> on Gd<sup>3+</sup> with bigger ionic radius [28]. Evidently, this is not the only mechanism. Absence of notable suppression of afterglow in LYSO:Ce when average ionic radius also increases with  $Y^{3+}$  addition presumes specific impact of Gd<sup>3+</sup> ions. For example, electron capture by traps can compete with migration of electrons via Gd<sup>3+</sup> sublattice, by the analogy with GSO:Ce [41]. Absence of afterglow in GSO:Ce despite the presence of silicon-non-bound oxygens evidences in favour of this suggestion.

LGSO is characterized by double-exponent luminescent decay under γ-irradiation. Alongside with fast component with decay time around 30-35 ns, slower component with  $\tau$  around 100 ns appears, which is associated with CeO<sub>6</sub> centers. Our data [28] certify the increase of slower component contribution from 2 to 18 % as Gd concentration increases from 25 to 80 %. Much more large contribution of slow component (8-56%) was reported in LGSO (20 % Lu) [39] certifying an activator redistribution to CeO<sub>6</sub> polyhedra. The obtained low afterglow level in LGSO:Ce at decrease of light yield only by ~20 % in comparison with LSO:Ce makes this material attractive for applications in introscopy where signal/noise ratio is a crucial parameter for obtaining good image contrast. In particular, works on obtaining LSO and LGSO films on LSO and YSO substrates for X-ray scintillation screens have been started [42]. Properties of the obtained crystals in comparison with known analogs are presented in the Table.

Table. Comparative table of scintillation characteristics of Ce doped rare earth orthosilicates.

Crystal	Light yield, Phot/MeV	Energy resolution (662 KeV), %	Decay time,	Afterglow level after 3 ms, %	Reference
GSO	8000	9-11	56, slow	0.040	[1], this work
LSO	25000	7.3 - 9.7	40	0.59	[1, 43]
LYSO	23000-25000	8-14	41	0.52	[43, 44]
GYSO	7000-9000	8-10	No data	0.020-0.037 (after 5 ms)	[19]
LGSO (20 % Lu)	15000 - 20000	6.5 - 10	46/114	5.6	[36], [41]
LGSO (90 % Lu)	20000-25000	8.0-9.8	40	No data	[45]
LGSO (30-60 % Lu)	20000-25000	6.9 - 7.3	31	0.075 - 0.145	This work

### 4. Conclusions

Three concentration ranges in LGSO:Ce solid solutions can be noted on the base of the complex study of scintillation characteristics:

- LSO and LGSO crystals (Lu concentration >60 at.%) demonstrate light yield around 25000 phot/MeV, high afterglow level, energy resolution around 8-9 % (662 KeV);
- moderate light yield near  $10000~\rm phot/MeV$  and energy resolution 9-13~% is observed in GSO and LGSO with GSO-type structure (Lu concentration in host  $<20~\rm at.~\%$ );
- optimal combination of scintillation characteristics was obtained in the middle concentration range (20-60 at.% of Lu). These crystals possess light yield 20000-25000 phot/MeV, energy resolution 6.9-7.3 %, afterglow level is not more than 0.15 % (after 3 ms). Contribution of slow decay component in the given concentration range not exceeds 2-3.5 %.

The obtained characteristics certify that LGSO:Ce is a attractive oxide scintillator for devices of medical diagnostics (PET), high-energy physics, other applications. Further efforts will be directed to optimization of growth procedures for these crystals aimed at minimization of inclusions and improvement of optical transmission and light yield. The parallel task is an increase of size of grown crystals and minimization of crack appearing. Advanced luminescent studies aimed at understanding of energy transfer processes in the given systems are under way.

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## Сцинтиляційні кристали твердих розчинів оксиортосилікатів

### О.Сідлецький

Розглянуто структуру, механічні, оптичні та сцинтиляційні властивості оксидних сцинтиляторів на основі твердих розчинів оксіортосилікатів рідкісноземельних елементів. Характеристики розроблених кристалів лютецій-гадолінієвого силікату  $Lu_{2-2x}Gd_{2x}SiO_5$ , активованого церієм, співставлені з кращими світовими аналогами. Властивостями отриманих кристалів є зниження рівня післясвітіння у декілька разів та зменшення внеску повільного компонента загасання сцинтиляцій. Обговорюються перспективи подальших робіт з оптимізації характеристик даного класу сцинтиляторів.