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Lu₃Al₅O₁₂-based materials for high 2D-resolution scintillation detectors

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ABSTRACT

About 20 μm thick Ce-doped Lu₃Al₅O₁₂ thin films grown by Liquid Phase Epitaxy and thin plates of similar thickness prepared by mechanical cutting and polishing from Czochralski grown crystals are used in 2D-imaging experiment down to μm 2D-resolution. Their scintillation response is also measured under α -particle excitation and performance of film and bulk material is mutually compared. Furthermore, scintillation and thermoluminescence characteristics of UV emitting Sc-doped LuAG grown by Czochralski method are presented since this system is a candidate material for UV emission-based 2D sensors with improved diffraction limit with respect to the presently used Ce-doped aluminum garnets.

Keywords: Thin scintillator films, 2D imaging, Ce-doped Lu₃Al₅O₁₂ (LuAG), liquid phase epitaxy growth, thermoluminescence

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1. INTRODUCTION

Some inspection/imaging applications require thin (few μm – tens of μm) and large area (up to 50 mm diameter) single crystalline plate or film scintillators [1–3]. Coupled with position sensitive photodetectors (CCD) they enable 2D-imaging of tiny (hidden) objects down to sub- μm scale under x-ray or accelerated electron exposure. Contradictory demands for high x-ray attenuation in the scintillator (requiring larger thickness) and excellent 2D-resolution (smallest possible plate/film thickness) call for using high density materials.

Ce³⁺ or Pr³⁺ doped and Lu-based compounds are under intense research as they can reach high density together with an excellent scintillation figure-of-merit [4, 5]. Lu₃Al₅O₁₂:Ce (LuAG:Ce) has been reported recently as highly efficient scintillator material, the scintillation efficiency of which can reach up to 700 % of BGO under x-ray excitation [6, 7]. LuAG host offers almost 50 % higher density ($\rho = 6.67 \text{ g/cm}^3$) and significantly larger effective atomic number $Z_{\text{eff}} = 59$ with respect to YAG ($\rho = 4.6 \text{ g/cm}^3$, $Z_{\text{eff}} = 29$). Bulk single crystals are grown by Czochralski technique from melt while single crystalline films are obtained by the Liquid Phase Epitaxy (LPE) method [8].

In this contribution we demonstrate the 2D-imaging ability of LuAG:Ce LPE-grown films and compare them with a Czochralski-grown plate of the same thickness ($\sim 20 \mu\text{m}$). Time dependence of scintillation response related to the delayed recombination phenomena, are discussed in the light of possible application in dynamic fluoroscopy imaging. Finally, we present technologically feasible modifications to obtain an UV-emitting material based on LuAG host by Sc doping which can lower the diffraction limit and enhance the 2D-resolution accordingly.

2. METHODOLOGY

Four single crystalline films (SCF) of (Lu-Y)AG:Ce grown by LPE from melt-solution based on PbO-B₂O₃ flux onto (110)-oriented YAG (quarter-moon of 20 mm diameter) and two LuAG:Ce plates of 20 μm thickness and 20 mm

diameter prepared from Czochralski grown bulk single crystals and glued on BK7 glass substrate were used in the imaging experiments, see Table 1 and Fig. 1. Furthermore, the 0.5wt% Sc-doped (in melt) bulk LuAG crystal was prepared and plate of about 7x7x1 mm and bulk of about 10x10x10 cm, all polished, were used for another spectroscopic experiments.

2D-imaging experiment is composed of microfocus X-ray source, sample holder and digital CCD camera with high 2D-resolution, see Fig. 2.

Sample acronym	Sample type	Film/plate material	Film/plate thickness [μm]	Substrate thickness [mm]	Scintillation efficiency [%]
Lu-6	LPE SCF	LuAG:Ce	17	0.5	70
Lu-9	LPE SCF	LuAG:Ce	29	0.5	67
Lu-11	LPE SCF	$\text{Lu}_{0.8}\text{Y}_{0.2}\text{AG:Ce}$	15	0.5	61
Lu-16	LPE SCF	$\text{Lu}_{0.6}\text{Y}_{0.4}\text{AG:Ce}$	27	0.5	62
Cz-2	Bulk Czochral	LuAG:Ce	20	3	100
Cz-3	Bulk Czochral	LuAG:Ce	20	3	92

Table 1. Characteristics of the samples used for the 2D imaging experiment.

Fig. 1 Visual appearance of LPE SCF samples Lu-6, 9, 11, 16 after polishing-off the film from one side and 5 μm thickness from the other side. On the right the Cz-2 sample is displayed with the LuAG:Ce plate on the top surface.

Fig. 2 2D X-ray imaging experiment. X-ray source is on the left.

In the imaging experiment, a sample from Table 1. was placed on the sample holder with the active layer/plate towards the X-ray source. An imaged object was placed between X-ray source and the sample. During the X-ray exposition (500 ms) the luminescence image from the scintillator sample is collected by the CCD camera. Expositions without any object were made as well to compare both the scintillation efficiency and film/plate homogeneity of the samples.

Scintillation photoelectron yield and its dependence on shaping time of the detection electronics were also measured using modern HPMT-based detector, for the experimental details see [9]. Radioluminescence spectra and scintillation decays were measured at modified Spectrofluorometer 199S; thermally stimulated luminescence (TSL) glow curves and emission spectra below and above RT were measured by a home-made set-up [5-7]. Excitation spectra in VUV spectral region were measured using synchrotron radiation at Superlumi station, HASYLAB, DESY, Hamburg.

3. RESULTS AND DISCUSSION

3.1 Optical quality and scintillation efficiency of the samples prepared for 2D imaging

In Fig. 3 an optical image of one of the LPE SCF samples and that of Cz-3 is provided using an optical microscope. Samples were back-illuminated with UV light to induce Ce^{3+} luminescence. In SCF samples there are occasional inclusions and surface flaws which scatter the luminescence light out from the sample, i.e. they appear as bright spots. Plates prepared from Czochralski grown bulk crystals are very homogeneous and do not show any scattering effects.

LPE SCF Sample Lu-9

Czochralski plate sample Cz-2

Fig. 3 Optical image of the samples back-illuminated with UV light. Magnification 57x.

In the case of X-ray excitation, the same effect is observed from the back side of the samples using the CCD camera: surface flaws in LPE-grown samples appear as bright spots, while Czochralski grown plate shows very homogeneous and spot-free image, Fig. 4.

Sample **Lu-6** **Lu-9** **Lu-11** **Cz-2**

Fig. 4 CCD camera image of X-ray irradiated samples. Bright spots are due to the surface flaws.

In the measurement displayed in Fig. 4 also the integrated emission intensity from the sample was evaluated from an average of 25 times repeated expositions and was used to evaluate scintillation efficiency of the samples, which is given in Table 1. normalized with respect to Cz-2 sample. The best from LPE SCF samples showed about 70% efficiency with respect to that of Cz-2 one.

3.2 2D imaging experiment

To determine 2D resolution of the examined scintillation sensors the CCD camera was equipped with additional focusing and enlarging optics. The 8 μm thick golden wire grid, see Fig. 5, was placed in front of the sample towards the X-ray source.

Fig. 5 SEM image of the golden wire grid. Thickness of wires is 8-11 μm .

Fig. 6 The grid image obtained by Lu-6 sample screen (upper left) with the detail of wire crossing (lower left) and the same picture obtained by Cz-3 sample (right part of the figure)

The above mentioned flaws in LPE SCF sample degrade the image quality with respect to that obtained by Czochralski grown plates, but the 2D-resolution appears in both groups of the samples practically the same as can be seen from the detail of wire crossing in the bottom part of Fig. 6. The same 2D-resolution is also evidenced from the output signal dependence across the grid wire, Fig. 7 (X-axis in CCD chip pixels, 1 pixel=0.74 μm). Better signal-to-background ratio from CCD camera by factor 1.5 is achieved in Cz-3 sample due to its higher scintillation efficiency.

Fig. 7 Electrical output signal from CCD pixels across the grid wire. Theoretical shape of the signal is also given.

It is worth noting that in the case of LPE SCF samples the output luminescence signal could be “contaminated” by the intrinsic emission of undoped LuAG substrate due to penetrating X-irradiation which can worsen the 2D resolution in the image due to defocusing effects. However, this intrinsic luminescence occurs within 300-350 nm [10] and is thus mostly

absorbed by the glass focusing optics in front of CCD camera. Moreover, a CCD chip shows lower detection efficiency in this spectral region with respect to the green region of the spectra.

3.3 Photoelectron yield and its time dependence under α -particle excitation

Basic scintillation properties (scintillation response) as photoelectron or light yields ($N_{\text{phels}}(E)$ or $LY(E)$, respectively), energy resolution (FWHM) and proportionality of the yields can be obtained from measurements of pulse height spectra of scintillating materials [11]. Recently, a new technique using hybrid photomultipliers (HPMT) is applied to measure scintillation response of Lu- or Y-based garnet compounds [12]. This technique allows to provide precise photoelectron calibration of the X- or γ -ray excited pulsed height spectra of scintillators [11-13]. Besides Ce^{3+} dopant also Pr^{3+} one was studied in LuAG or YAG and results are given in detail in [5,14]. Nowadays, LuAG:Ce crystals are characterized by (i) light yield around 15.000,- ph/MeV, (ii) energy resolution $\sim 10\%$ at 662 keV and (iii) photoelectron yield proportional in the energy range 500-1300 keV. However, a deviation from proportionality (the decrease of about 30%) is observed in the energy range 500-10 keV.

Characterization of scintillation properties of SCF requires somewhat modified experiment in the scintillation response measurements. Due to small SCF thickness (from a few μm to 60 μm) it is not possible to use γ -ray excitation because of small γ -ray absorption in the SCF itself and prevalent energy deposit in the substrate. In such a case the emitted light would consist of that generated in the SCF film (Ce^{3+} emission) and the dominant UV emission coming from a thick substrate. The only possibility to characterize and compare SCF scintillation response is the use of α -ray excitation (e.g. 5.4857 MeV α -rays of ^{241}Am) [9]. According to detailed calculations, the penetration range of alpha-particles in LuAG and YAG crystals varies from 7 to 10 μm , respectively [15]. The α -rays excited scintillation response – pulse height spectra can be measured by the same set-up used for γ -ray excitation [11-13] using the α -ray sources (radioisotopes) placed directly onto the sample surface.

N_{phels} photoelectron yield (pulse height spectra) of LuAG:Ce and LuYAG:Ce SCF (samples Lu 6, 9, 11 and 16, see Fig. 1) were measured under ^{241}Am α -particle excitation (5.4857 MeV) in the shaping time range 0.5 – 10 μs together with two LuAG:Ce single crystals (SC 1 and 2 cut from the same parent boule as Cz-2 and Cz-3 samples above). Results of measurements are summarized in Fig. 8 and Table 2.

Fig. 8 The dependence of normalized photoelectron yield on the shaping time under α -rays excitation of ^{241}Am (energy 5.4857 MeV). The lines are logarithmic fits.

Sample acronym	Sample type Film/plate thickness	Film/plate material	$N_{\text{phels}}/\text{MeV}$ (phels) under ^{241}Am at 1 μs shaping time	FWHM (%) at ^{241}Am at 1 μs shaping time	Difference $N_{\text{phels}}/\text{MeV}$ (in %) between 0.5 and 10 μs shaping times
Lu-6	SCF (17 μm)	LuAG:Ce	1252	10.5	20.8
Lu-9	SCF (29 μm)	LuAG:Ce	1063	16.9	16.8
Lu-11	SCF (15 μm)	$\text{Lu}_{0.8}\text{Y}_{0.2}\text{AG:Ce}$	838	11.2	17.3
Lu-16	SCF (27 μm)	$\text{Lu}_{0.6}\text{Y}_{0.4}\text{AG:Ce}$	837	17.6	6.8
SC 1 (Cz-2)	Bulk (1 mm)	LuAG:Ce	2136	9.1	68
Sc 2 (Cz-3)	Bulk (1 mm)	LuAG:Ce	2026	8.9	79.3

Table 2 Scintillation response properties of measured LuAG:Ce, LuYAG:Ce SCF samples compared with those of CZ grown LuAG:Ce single crystals ($N_{\text{phels}}/\text{MeV}$ photoelectron yield, FWHM and $N_{\text{phels}}/\text{MeV}$ difference (in %) between 0.5 and 10 μs shaping times).

From Fig. 8 and Tab. 2 we can clearly see that Czochralski grown LuAG:Ce crystals exhibit much more slow components (about 60 %) than LuAG:Ce or LuYAG:Ce SCF samples, see also last column of Tab. 2. However, the photoelectron yield $N_{\text{phels}}/\text{MeV}$ of LuAG:Ce crystals is distinctly higher than that of Ce-doped LuAG or LuYAG SCF samples.

Fig. 9 Radioluminescence (RL) spectrum of 0.5%Sc-doped LuAG in absolute comparison with standard BGO scintillator sample. RT, ex citation by X-ray tube, 40 kV, 15 mA. Ex citation spectra of intrinsic luminescence of undoped LuAG (em=330nm) and that of Sc-related one (em=275 nm).#

3.4 UV emitting LuAG-based scintillators

It has been already mentioned in the literature that doping by isovalent ions of sufficiently different radius at Lu (Y) or Al site in aluminum garnets will result in appearance of UV emission bands [8,16-20]. The nature of such bands is usually interpreted in terms of localized (trapped) excitons around such substituted sites [18-20]. Scintillation characteristics of the Sc-doped LuAG single crystal [19] and SCF films [20] have been already reported in the literature. Radioluminescence spectrum of 0.5%Sc-doped LuAG is in Fig. 9. UV emission band shows the maximum at about 275 nm and an extended tail towards 400 nm can be interpreted as a residual intensity of the intrinsic undoped LuAG host emission in agreement with [19,20]. The 275 nm band is interpreted as a trapped exciton around the Sc-embedded site [19]. Excitation spectrum of the 275 nm emission closely follows that of the intrinsic 330 nm emission of undoped LuAG, see Fig. 9, which supports such an interpretation further. Scintillation decay reported in Fig. 10 is approximated by a two-exponential fit with the decay times of about 5 ns and 1330 ns, where the latter component contains more than 95% of the emitted scintillation light. TSL glow curve and spectra are provided in Fig. 11. It is interesting to note great similarity of the glow curve with those of undoped LuAG samples [21] where the glow curve was interpreted in terms of thermally induced liberation of holes from unspecified traps and their recombination with electrons localized elsewhere. In Fig. 11 the band at 2 eV is the second order of the UV emission. The similarity of TSL and radioluminescence spectra in Fig. 9 and 11 suggests that electrons are localized around Sc ions sites and thermally liberated holes recombine with them. Due to the absence of intense TSL peaks above 130 K the afterglow of such a system at RT in millisecond time scale is expected to be rather low in accordance with the data provided in [19]. The growth of Sc-containing LPE films has been reported in [8], but the films were further codoped with Ce so that the dominant emission was that of Ce^{3+} in the green region of spectra. It is further expected that scintillation efficiency of such UV emitting $\text{Lu}_3(\text{Al},\text{Sc})_5\text{O}_{12}$ films will be decreased due to the use of PbO-based flux [22] so that other fluxes based e.g. on BaO would be necessary to reach good scintillation efficiency of such a UV emitting Sc-doped LuAG SCF. Due to the fact that the emission band of this system is peaking at 275 nm, it is expected that in the 2D-imaging application the diffraction limit can be decreased by almost a factor 2 with respect to the Ce-doped films used in the imaging experiment above.

Fig. 10 Spectrally unresolved scintillation decay of 0.5%Sc-doped LuAG at RT. Excitation by ^{22}Na radioisotope (511 keV). The fit by solid line is the convolution of instrumental response and the function $I(t)$ in the figure.

Fig. 11) TSL glow curve (obtained from integration of a wavelength resolved measurement in the 200-850 nm interval) after X-ray irradiation at 10 K of 0.5%Sc-doped LuAG. b) TSL emission spectra within the specified temperature regions of the glow curve displayed in (a). Spectra are shifted on the ordinate scale for better clarity.

4. CONCLUSIONS

Using microfocused X-ray source and high resolution CCD camera with the appropriate focusing optics the 2D imaging with thin LPE films or plates (both about 20 μm thick) of LuAG:Ce single crystal can achieve sufficient contrast and the 2D resolution about 1 μm . Scintillation efficiency and photoelectron yield from present LPE films is somewhat inferior to those of single crystal plate of the same thickness made from Czochralski grown material. The deterioration of the former could be partially due to the use of PbO-based flux as already noted in the literature [23]. LuAG host offers a possibility to prepare an efficient UV emitting scintillation sensor with the emission peak at 275 nm when Sc doping is used at the level of 0.5%. 2D sensor based on such a material is expected to have the diffraction limit of about a factor of 2 lower with respect to presently used Ce-doped aluminum garnets.

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