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# Growth and scintillation properties of Sc, Pr, Ce co-doped LuAG epitaxial garnet layers

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Abstract. Single crystalline Sc, Pr, Ce co-doped Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> garnet layers were grown by liquid phase epitaxy. The Sc doping increases the scintillation response of Pr<sup>3+</sup> activator ions because of overlap of the Sc-related emission around 275 nm with the 4f $\rightarrow$ 5d absorption band of Pr<sup>3+</sup> centers. The radioluminescence (RL) spectra give evidence for presumed energy transfer form Sc<sup>3+</sup> to Pr<sup>3+</sup> activator ions. Significant increase of the RL emission output was observed when co-doping with both Pr and Sc ions compared to Sc-free samples. The maximum integral RL was noticed for samples with relatively high Sc concentration, 1.2 - 3 at %. On the contrary, an increase of the photoelectron yield, N<sub>phe</sub>, measured under alpha particle excitation, was observed only for rather low Sc content, ~0.6 at %, at higher Sc concentrations N<sub>phe</sub> decreased. Nevertheless, LuAG epitaxial layers with optimal Sc and Pr contents show comparable or even a bit higher N<sub>phe</sub> yield and energy resolution than Cz-grown Pr:LuAG single crystals. Furthermore, the slow components in photoelectron response were significantly suppressed in epitaxial layers compared to single crystals.

# 1. Introduction

 $Pr^{3+}$ -doped aluminum garnets or perovskites exhibit broad emission in the ultraviolet spectral range and very fast luminescence in the nanosecond range. This is due to the fact, that the lowest <sup>2</sup>D crystal field split states of the 5d configuration are positioned at lower energies than  ${}^{1}S_{0}(4f^{2})$  state. Under high energy excitation of Pr-doped LuAG or YAG, a wide band emission is observed in the ultraviolet spectral range between 300 and 400 nm. It originates from the parity allowed interconfigurational 5d -4f transitions. Due to high intensity and short decay time ~ 20 ns of this emission, these systems are potential candidates in fast scintillation applications [1].

Doping of yttrium or lutetium aluminum garnets with isovalent  $Sc^{3+}$  ions, which occupy preferably the aluminum octahedral sites, induces an intense broad emission band near 275 nm in photoluminescence (PL) or in radioluminescence (RL) spectra [2]. The  $Sc^{3+}$  ions have closed shell configuration and they do not show any emission in the visible and UV spectral regions. However,

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they induce defects in the garnet lattice due to different ionic radii of  $Sc^{3+}$  and  $Al^{3+}$  ions,  $r_{VI}(Sc) = 74.5$ and  $r_{VI}(Al) = 53.5$  pm. This emission, with the decay time ~ 600 ns, originates from recombination of an exciton located near this defect. The lowest absorption band of  $4f \rightarrow 5d$  allowed transition of  $Pr^{3+}$ ions is situated at 280 nm, i.e. just at  $Sc^{3+}$  emission, and energy transfer from Sc to Pr activator ions can be expected. Therefore co-doping of garnets by  $Sc^{3+}$  ions may additionally increase the emission output from the  $Pr^{3+}$  ions via excitation energy transfer to these ions due to good spectral overlap of Sc-emission and Pr-absorption spectral bands. An attempt with ScPr doping was made by Dorenbos et al [3] only for fixed Sc concentration of 1 at % but any positive effect of Sc co-doping was not reported.

The goal of this work was to study the effect of  $Sc^{3+}$  co-doping on the emission properties of  $Pr^{3+}$  ions in wide range of Sc concentrations from 0.025 to maximum possible 10 at % in order to find out optimal Sc and Pr concentrations providing maximal light yield. We studied in detail the PL and RL properties, and the photoelectron yield excited by alpha particles. Several samples were co-doped also by Ce<sup>3+</sup> ions which have intense emission in the green-yellow spectral range.

# 2. Samples

The single crystalline garnet layers were grown by the isothermal dipping liquid phase epitaxy from the flux onto Czochralski grown YAG and LuAG substrates of (111) crystallographic orientations and 20 mm in diameter. Most of layers were grown onto LuAG substrates, layers with higher Sc content, > 2 at %, were grown onto YAG substrates, which match better the lattice constant. The layers were grown in the Pt crucible from supercooled melts at a constant supercooling. Starting raw materials of 5N purity were used.

All the epitaxial layers were grown under nearly the same growth conditions, i.e. growth temperature, supercooling, growth rate, and the solvent composition, in order to eliminate the effect of growth conditions on the sample properties. The growth temperatures were  $1025 \pm 5$  °C, the supercooling was 10 - 20 K. The layer thickness, determined by weighing, was 6 - 18 µm, most of films have the thickness around 10 µm.

Several sets of undoped, Sc-doped, Pr-doped, ScPr co-doped, and ScPrCe co-doped LuAG layers were grown with various Pr and Sc contents. In the technology process we used a novel lead-free BaO-B<sub>2</sub>O<sub>3</sub>-BaF<sub>2</sub> flux recently developed for growing of aluminum garnets [4]. Basic advantage of BaO-based flux, compared to the routinely used PbO flux, is possibility to obtain layers with high transparency and with negligible content of impurity ions. Scintillator systems with efficient UV emission down to 220 nm can be grown from the BaO flux. It is worth noting, that layers grown from routinely used PbO-B<sub>2</sub>O<sub>3</sub> flux exhibit increased absorption in the ultraviolet spectral range due to the impurity Pb<sup>2+</sup> and Pt<sup>4+</sup> ions present layers [5] and Pr-doped garnet or perovskite epitaxial layers grown from the PbO flux show decreased partly quenched UV emission [6,7].

The composition of layers was determined by electron-probe and GDMS microanalyses. Unlike activator Pr and Ce ions, which were doped into the garnet lattice in concentrations of the order of several hundreds ppm, the scandium atoms were substituted in rather high concentrations up to 10 at %, corresponding to hypothetical formula  $Lu_3Sc_2Al_3O_{12}$ . We note, that some part of Sc ions occupy also dodecahedral Lu sites due to relatively high ionic radius of Sc,  $r_{VIII}(Sc) = 87$  pm,  $r_{VIII}(Lu) = 97.7$  pm [8]. The X-ray diffraction measurements demonstrate high crystallographic quality of layers quite comparable with that of substrates. The parameters of selected samples are displayed in Table 1. Concentrations of Sc, Pr, and Ce dopants are given in at % relative to the total number of atoms in  $Lu_3Al_5O_{12}$  molecule.

# **3.** Properties of epitaxial films

The optical properties were studied using the optical absorption, photoluminescence (PL) and radioluminescence (RL) spectroscopy in the spectral range from ultraviolet to the near infrared. The decay of PL was measured by the single photon counting method under excitation of nanosecond

**Table 1.** Characteristics and scintillation properties ( $N_{phe}/MeV$  photoelectron yield at 1 µs shaping time and FWHM, alpha particle excitation <sup>239</sup>Pu 5.16 MeV) of Sc:LuAG, Sc,Pr:LuAG and Sc,Pr,Ce:LuAG epitaxial layers and comparison with Pr:LuAG and LuAG single crystals.

| Sample          | No        | Thickness | Sc content | Pr content                 | $N_{phe}(1 \ \mu s)$ | FWHM |
|-----------------|-----------|-----------|------------|----------------------------|----------------------|------|
|                 |           | (µm)      | (at %)     | $(10^{-3} \text{ at } \%)$ | $(MeV^{-1})$         | (%)  |
| LuAG            | 1LB1      | 18.1      | -          | -                          |                      | 10.2 |
| Sc,Pr:LuAG      | 1LBS3     | 11.6      | 0.025      | 9.1                        | 427                  | 8.2  |
| ScPr:LuAG       | 1LBS7     | 5.5       | 1.8        | 9.1                        | 327                  | 32.8 |
| Sc,Pr:LuAG      | 1LBS8     | 6.9       | 3.95       | 9.1                        | 244                  | 12   |
| Sc,Pr:LuAG      | 1LBS10    | 5         | 10         | 9.1                        |                      |      |
| Sc,Pr:LuAG      | 3LBS2     | 6.3       | 0.6        | 1.2                        | 935                  | 8    |
| Sc,Pr:LuAG      | 3LBS4     | 11.7      | 0.6        | 4.4                        | 689                  | 9    |
| Sc:LuAG         | 4LBS2     | 5.7       | 0.14       | -                          | 591                  | 10.6 |
| Sc:LuAG         | 4LBS5     | 7         | 1.83       | -                          | 327                  | 32.8 |
| Sc,Pr:LuAG      | 5LBS4     | 12.3      | 2.9        | 4.8                        | 520                  | 15.7 |
| Sc,Pr,Ce:LuAG   | 5LBS6     | 10.9      | 2.9        | 4.8 +                      | 277.5                | 25.5 |
|                 |           |           |            | Ce 4.0                     |                      |      |
| Sc,Pr:LuAG      | 6LBS4     | 10.7      | 1.2        | 5.2                        | 680                  | 12.9 |
| Pr:LuAG         | 7LBS3     | 13.6      | -          | 8.5                        | 568                  |      |
| LuAG:Pr crystal | 8846/1    | 1 mm      | -          | 28                         | 584                  | 8.3  |
| LuAG:Pr crystal | 8849/1    | 1 mm      | -          | 46                         | 700                  | 10.7 |
| LuAG            | 3LBS1     | ~ 0.5 mm  | -          | -                          | 937                  | 12.7 |
| Substrate       | substrate |           |            |                            |                      |      |

hydrogen flash lamp. Scintillation response of epitaxial layers was studied under  $\alpha$ -particle excitation. All measurements were made at room temperature.

# 3.1. Optical absorption

Optical absorption of epitaxial layers was measured in the spectral range 190-1100 nm. Absorption coefficient of ScPrCe:LuAG layers for Sc doping level 0.6 at % and varying Pr content from 0 to 0.0082 at % is shown in figure 1.

Optical transparency of all epitaxial layers grown from the BaO flux is high and any impurity related absorption is not observed at wavelength between 1100 and 230 nm. The broad intense spectral bands at 240 and 282 nm come from parity allowed  $4f \rightarrow 5d (Pr^{3+})$  transitions to the two lowest crystal field split 5d states. The  $4f-4f(Pr^{3+})$  parity forbidden transitions are not discernible in the absorption spectra due to low doping levels and low thickness of layers. The doping of isoelectronic Sc<sup>3+</sup> ions does not induce any new features in the absorption spectra. A slight increase of absorption coefficient, which is apparent below 230 nm, presumably comes from certain decrease of a band gap of LuAG due to Sc doping and from potential unidentified impurities coming from the flux. The Sc doping has no obvious effect on the absorption coefficient related to the allowed 4f-5d transitions of rare earth ions, Pr and Ce.

# 3.2. Photoluminescence (PL)

The excitation and emission spectra of the PL were measured in the spectral range from 200 to 850 nm. The LuAG layers doped with  $Pr^{3+}$  ions show intense ultraviolet PL between 290 and 420 nm corresponding to allowed transitions from crystal field split 5d states to  $4f({}^{3}H_{J}, {}^{3}F_{J})$  manifolds, figure 2a. This  $Pr^{3+}$  emission has principal maximum at 308 nm whose position practically does not depend on Sc co-doping. Narrow intra-configurational  $4f \rightarrow 4f(Pr^{3+})$  transitions from  ${}^{3}P$  and  ${}^{1}D$  terms, observed near 480 and 615 nm, are very weak. Integral intensity of all f-f contributions is < 4 % compared to



**Figure 1.** Absorbance of ScPr:LuAG epitaxial layers grown on LuAG substrates with constant Sc content and increasing Pr content, in at %, specified in legend. LuAG single crystal substrate is also shown for comparison. The results were not corrected for reflection.

that of 5d $\rightarrow$ 4f spectral band. The peaks in the excitation spectra at 240 and 282 nm are closely related to the absorption ones. All the PL features of Pr:LuAG are practically identical as those observed in single crystal counterparts, cf. ref. [9 - 11].

The PL spectra of ScPr and ScPrCe co-doped LuAG films are shown in figures 2b and 2c, respectively. Any Sc related emission is not observed in the PL spectra (note that PL was excited at energies below the band gap). The most striking feature observed in the PL spectra is significant increase of PL intensities of forbidden f-f transitions at 480 and 615 nm. This increase linearly depends on Sc content and for 10 at % of Sc the relative intensity of f-f transitions is about an order of magnitude higher compared to Sc-undoped samples. The mechanism of energy transfer towards <sup>3</sup>P and <sup>1</sup>D terms is not obvious, but presumably nonradiative transitions from 5d states to these terms may play a crucial role.

Additional Ce<sup>3+</sup> doping, ScPrCe:LuAG samples, on the contrary, decreases the PL of Pr<sup>3+</sup> activator ions, sample 5LBS6 in figure 2c. Broad green-yellow  $5d \rightarrow 4f$  emission from the Ce<sup>3+</sup> ions observed between 480 and 650 nm is relatively weak. Substantial energy transfer from Sc or Pr to Ce ions is not obvious and emission quenching of 5d(Pr) emission due to Ce doping is rather observed.

# 3.3. Decay kinetics

In order to obtain more detailed information on the role of impurities in the PL emission of epitaxial garnet films, we measured the 5d  $\rightarrow$  4f PL decay kinetics of  $Pr^{3+}(4f^2)$  ions, figure 3. The samples were excited at 282 or 340 nm and the 5d emissions of  $Pr^{3+}$  or  $Ce^{3+}$  were collected at 320 or 520 nm, respectively. The deconvolution of decay curves enabled us to determine individual contributions in the PL decay. A detailed inspection shows two-components for 320 nm  $Pr^{3+}$  emission with dominant 18-19 ns decay and a weak ~80-100 ns component. This slow component was also observed in Pr-undoped layers and it likely originates from the host. Only one 55 ns component was observed for 5d  $Ce^{3+}$  emission at 520 nm. The obtained values of decay times are in close relation with those reported for high quality Cz single crystals [9]. The decay times are independent on Sc content in layers up to the highest Sc concentrations used. We can conclude that in layers grown from a lead-free BaO flux, the PL decay curves do not indicate any notable loss of energy.



**Figure 2.** Photoluminescence excitation ( $\lambda_{em} = 310 \text{ nm}$ ) and emission ( $\lambda_{exc} = 282 \text{ nm}$ ) spectra of ScPrCe co-doped LuAG epitaxial layers. Concentrations of dopants, in at %, are shown in the legend.

# 3.4. Radioluminescence (RL)

The RL of layers was measured under X-ray excitation, (10 kV, 50 mA) in the spectral range 200 - 670 nm. All the RL spectra were measured along with a reference BGO (Bi<sub>4</sub>Ge<sub>3</sub>O<sub>12</sub>) single crystal, which enabled us to estimate the relative X-ray yield and mutual comparison of samples.

The RL spectra of ScPr co-doped LuAG layers are displayed in figure 4. The observed basic features are following: first, the integral RL intensity is an order of magnitude higher compared to reference BGO crystal. Second, integral RL of ScPr co-doped LuAG increases in the spectral range 200 - 670 nm with growing Sc content, this increase is 60 % for 1.2 at % of Sc and 80 % for 2.9 at % of Sc. Comparing the RL spectra with those of LuAG doped either solely by Sc or solely by Pr ions, the main contributions from individual ions were easily identified and they are indicated by arrows in figure 4. The principal Sc<sup>3+</sup> and Pr<sup>3+</sup> - related peaks are situated in the RL spectra at 280 nm and 308 nm, respectively. In the PL spectra, see figure 3, the Sc emission at 280 nm was not excited. The most



**Figure 3.** Photoluminescence decay curve of ScPr co-doped LuAG epitaxial films measured at  $Pr^{3+}(5d\rightarrow 4f)$  emission,  $\lambda_{em} = 320$  nm,  $\lambda_{exc} = 282$  nm. The solid line is convolution of the fitting curve I(t) with the instrumental response.

remarkable feature observed for higher Sc concentrations, figure 4a, is that the relative intensity of Sc peak at 280 nm diminishes despite the fact that Sc content increases. Quite opposite tendency is observed for  $Pr^{3+}$  contribution – the peak at 308 nm significantly increases for increasing Sc content while the Pr content practically does not change. This notable dependence can be explained by the energy transfer from Sc to Pr ions. The Sc-related emission does not increase in proportion to Sc concentration and, on the contrary, the excitation energy is transferred from Sc sensitizer to Pr activator ions whose emission intensity increases. This dependence was observed for relatively high Sc concentrations, from 1.2 up to 3 at % of Sc. However, for even higher Sc concentrations > 3 at %, figure 4b, the RL intensity of 5d→4f band in the UV spectral range decreases due to presumed concentration quenching at high Sc content and continuing nonradiative transfer of energy to <sup>3</sup>P and <sup>1</sup>D terms. At 10 at % of Sc the RL intensity in the UV range is drastically reduced.

The RL spectra support the observations for the PL ones: the f-f emission at 480 and 615 nm also amplifies significantly compared to d-f emission. This indicates nonradiative relaxation from 5d states to  ${}^{3}P$  and  ${}^{1}D$  terms of  $Pr^{3+}$  ions.

The Ce<sup>3+</sup> doping of ScPrCe:LuAG samples decreases the RL intensity in a similar way as observed in the PL and emission quenching due to Ce doping is observed.

#### 3.5. Scintillation properties

Scintillation properties of Sc:LuAG, Sc,Pr:LuAG and Sc,Pr,Ce:LuAG epitaxial layers were measured under alpha-particle excitation (mainly using <sup>239</sup>Pu alpha particle source of energy 5.16 MeV). Experimental set-up was described elsewhere [12, 13]. This method uses hybrid photon detection and it consists of HPMT (DEP 475 model of S20 cathode sensitivity) and electronics (Ortec 672 Spectroscopy Amplifier, Ortec 927<sup>TM</sup> Dual Multichannel Buffer and PC) for pulse height spectra measurements [12,14].

The largest photoelectron yield,  $N_{phe}$ , was observed for ScPr:LuAG samples containing low Sc or Pr concentrations (~0.6 at % of Sc), figure 5a. Observed  $N_{phe}$  yield was higher up to 30 % (sample 3LBS2) or comparable as those of Cz-grown Pr:LuAG crystals (see also Table 1). The time dependence of the photoelectron yield in the shaping time range 0.5 – 10 µs shows, figure 5b, that ScPr:LuAG epitaxial layers have substantially smaller  $N_{phe}$  time difference (of about 20 %) compared to Pr:LuAG single crystals (where it is about 50 % and it is not still saturated). This means that the layers have smaller content of slow components, probably due to less content of antisite defects in epitaxial layers [15, 16]. However, at higher Sc concentrations, > 2 at %, fraction of slow components in layers again increases.

Another important scintillation property is energy resolution (FWHM). The FWHM of epitaxial layers and single crystals are comparable, their values are of about 10 % or even a bit less. Again, ScPr:LuAG samples of low Sc and Pr concentrations, which have the highest  $N_{phe}$  yield, have also better FWHM, ~ 8 %. The Ce doping leads to the decrease of the  $N_{phe}$  even more and the emission is



**Figure 4.** Radioluminescence spectra of ScPr co-doped LuAG epitaxial layers: (a) samples with low and medium content of Sc, (b) heavily Sc-doped samples. Concentrations of Sc and Pr, in at %, is shown in the legend, Pr content is approximately the same in individual panels. Arrows indicate respective contributions originating from Sc<sup>3+</sup> and Pr<sup>3+</sup> ions. The spectrum of BGO crystal is shown for comparison.

quenched in a similar way as in the RL spectra.

We can conclude that epitaxial LuAG layers grown from the BaO flux have competitive scintillation properties with single crystals. The best photoelectron yield was obtained for low Pr, Sc concentrations. Such samples are comparable with Pr:LuAG single crystal counterparts. Furthermore, the fast components are notably higher in layers than in Cz-grown crystals, and the slow components are rather suppressed in layers: they are < 20 % for 0.6 at % of Sc, < 25 % for 1.2 at % of Sc, but it is ~35-45% for 2.7 at % of Sc. In single crystals the slow components are between 40 and 60 %. At even higher Sc concentrations, > 3 at % of Sc, the N<sub>phe</sub> yield significantly decreases and the energy transfer is not observed in scintillation properties.



**Figure 5.** (a) Photoelectron yield,  $N_{phe}$  per 1 MeV, at different shaping times under alpha particles (<sup>239</sup>Pu) excitation of ScPr:LuAG epitaxial layers, (b) time development of  $N_{phe}$  normalized to the shortest shaping time 0.5 µs for the same samples. Concentrations of Sc and Pr, in at %, are shown in the legend. Czochralski grown single crystal Pr:LuAG, sample 8849/1, is shown for comparison. The lines are guides for eye.

# 4. Conclusions

ScPr co-doped Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> garnet layers were grown by liquid phase epitaxy. The increase in radioluminescence compared to Sc-free sample was attributed to the energy transfer from Sc<sup>3+</sup> to Pr<sup>3+</sup> activator ions. The maximum integral RL was observed in samples with relatively high Sc concentration, 1.2 - 3 at %, and it was almost two times higher compared to samples without Sc codoping. However, the results for photoelectron yield, N<sub>phe</sub>, measured under alpha particle excitation are to some extent contradictory. Improvement of N<sub>phe</sub> was observed only for rather low Sc content, 0.6 at %, and Pr content ~0.002 at %, at higher Sc concentrations N<sub>phe</sub> yield decreased. This can be partly explained by the fact that long components in the ms time scale (e.g. contributions from forbidden f-f transitions) are also detected in the RL measurements, but the shaping time for photoelectron measurements was 10 µs at maximum. In spite of that, LuAG epitaxial layers with optimal Sc and Pr concentrations show even higher N<sub>phe</sub> yield than Cz-grown Pr:LuAG single crystal counterparts. Another remarkable result is that the slow components in photoelectron response are significantly suppressed in optimized ScPr doped layers compared to Pr:LuAG single crystals. However, intensity of slow components increases again for higher Sc content, > 3 at %. Furthermore, the energy resolution of ScPr co-doped layers is comparable or even a bit better than that of single crystals.

Increase of slow component in photoelectron yield with increasing Sc content can be tentatively explained by new stable electron traps, which were induced by the presence of Sc ions. These traps would delay radiative recombination at  $Pr^{3+}$  ions. They may have similar origin as relatively deep traps found in ScCe co-doped bulk crystals [17, 18].

We can conclude that Sc, Pr co-doped LuAG crystals with optimized dopant content can provide in many aspects better scintillation response compared to samples without Sc co-doping.

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