Effect of Al and Ce Ion Concentrations on the Optical Absorption and Luminescence in Gd₃(Al,Ga)₅O₁₂:Ce³⁺ Epitaxial Films

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Abstract—We have studied the effect of Al and Ce ions on the optical absorption and luminescence of single-crystal (Pb,Gd) $_{3-y}$ Ce $_y$ Al $_x$ Ga $_{5-x}$ O $_{12}$ (x=2.02, 2.09, 2.13, 2.17, 2.22; <math>y=0.02, 0.06, 0.07) films grown on (111)-oriented single-crystal Gd $_3$ Ga $_5$ O $_{12}$ substrates by liquid-phase epitaxy from supercooled high-temperature solutions using solvents of the PbO-B $_2$ O $_3$ system and growth charges containing 2.0, 2.1, or 2.2 mol % aluminum oxide and 0.03 or 0.2 mol % cerium oxide. The shift of the absorption bands of the Ce $^{3+}$ 5 d_1 and 5 d_2 levels has been determined as a function of Al concentration in the films. The intensity of the Ce $^{3+}$ luminescence bands of the films has been shown to increase with increasing Al and Ce concentrations.

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INTRODUCTION

This paper presents a continuation of a previous study [1] which showed that, in $Gd_3Al_xGa_{5-x}O_{12}$: Ce^{3+} epitaxial films grown on single-crystal gadolinium gallium garnet (GGG), Gd₃Ga₅O₁₂, substrates by liquidphase epitaxy (LPE) from supercooled high-temperature solutions using solvents of the PbO-B₂O₃ system and growth charges containing 1, 1.5, or 2 mol % Al₂O₃ and 0.03 or 0.2 mol % CeO₂, increasing the aluminum content to x = 1.9 shifted two absorption bands of the Ce^{3+} ion. Its $5d_2$ level shifted by 5 nm, from 346 nm (peak absorption wavelength) in a $Pb_{0.02}Ce_{0.03}Gd_{2.95}Ga_5O_{12}$ (1 at % Ce^{3+}) film to 341 nm in a $Pb_{0.03}Ce_{0.06}Gd_{2.91}Al_{1.9}Ga_{3.1}O_{12}$ (2 at % Ce^{3+}) film, and its $5d_1$ level shifted from 426 to 438 nm (by 12 nm). The intensity of the Ce³⁺ luminescence band centered at 550 nm was shown to increase eight times with an increase in Al content from x = 1.1 to 1.9 [1], and the band gap of the films was shown to increase by 0.2 eV. In addition, Vasil'eva et al. [1] demonstrated a systematic increase in luminescence intensity with increasing excitation photon energy in a wide spectral range, from the UV to soft X-rays (220 eV), which points to efficient high-energy radiation conversion into luminescence.

Interest in the $Gd_{2.97}Ce_{0.03}Al_2Ga_3O_{12}$ (1 at % Ce^{3+}) material has been aroused by the finding that it offers a considerable light output (up to 46000 photons/MeV), good energy resolution (4.9% at 662 keV), and high density (6.63 g/cm³) and is neither radioactive nor hygroscopic [2]. Czochralski-grown Gd_{2.97}Ce_{0.03}Al₂Ga₃O₁₂ crystals are used as X-ray, gamma-ray, alpha-particle, beta-particle, and neutron detectors and in medical imaging devices [3]. The optical properties of this material continue to be the subject of research with the aim of understanding how they vary with the Ga/Al concentration ratio, gaining greater insight into the processes underlying excitation energy conversion into a scintillation response, and assessing the effect of luminescence quenching on these processes [4–6].

The purpose of this work was to compare the optical properties of single-crystal $Gd_3(Al,Ga)_5O_{12}$:Ce films grown from high-temperature solutions with higher aluminum oxide concentrations in comparison with previous work [1] (2.0, 2.1, and 2.2 mol %) at the same cerium oxide concentration in the growth charge (0.03 and 0.2 mol %).

High-temperature solution	mol % Al ₂ O ₃	mol % CeO ₂	R_1	R_2	δt,°C	h _{max} , μm	$f_g^{ m max}, \ \mu m m/min$
I	2.0	0.03	35.42	15.71	985–962	15.9	0.37
II	2.1	0.03	35.81	15.69	968–964	30.4	0.34
III	2.2	0.03	36.19	15.67	969–964	17.0	0.24
IV	2.0	0.2	15.35	15.68	939–934	17.0	0.28
V	2.1	0.2	15.52	15.66	956–937	20.9	0.34
VI	2.2	0.2	15.68	15.64	957—946	19.6	0.51

Table 1. Growth charge composition and growth parameters of the Gd₃(Al,Ga)₅O₁₂:Ce epitaxial films

 δt is the temperature range where the saturation temperature was determined

EXPERIMENTAL

 $Gd_3(Al,Ga)_5O_{12}$:Ce epitaxial films were grown on (111)-oriented single-crystal GGG substrates by LPE from supercooled high-temperature solutions using solvents of the PbO- B_2O_3 system and growth charges containing 2.0, 2.1, or 2.2 mol % Al_2O_3 and 0.03 or 0.2 mol % CeO_2 (Table 1). The gadolinium oxide content in all of the high-temperature solutions was 0.2 mol %. The composition of the growth charge for the preparation of the epitaxial films had the following molar ratios R_1 and R_2 of their components (Table 1):

$$R_1 = ([Ga_2O_3] + [Al_2O_3])/([Gd_2O_3] + [2CeO_2]),$$

 $R_2 = [PbO]/[B_2O_3].$

The growth charge was prepared from the following oxides: reagent-grade PbO (D/SPA), OSCh 11-3 B_2O_3 , GdO-1 Gd_2O_3 , TseO-SS CeO_2 , OSCh 15-2 Ga_2O_3 , and analytical-grade Al_2O_3 .

We grew 25 film—substrate—film samples 10 \times 15 mm in dimensions using six batches of freshly prepared high-temperature solutions (Table 1), as described by Vasil'eva et al. [7]. The growth time of the epitaxial films at various supercoolings was 5, 10, 30, 60, 75, 80, 90, or 120 min. The substrate rotation rate was 50, 100, 124, or 132 rpm. For each batch of high-temperature solutions, we determined the maximum thickness of the films, $h_{\rm max}$, and the maximum growth rate observed in our experiments, $f_{\rm g}^{\rm max}$ (Table 1). It is worth pointing out that, as the Al₂O₃ content of the

growth charge was increased from 2.0 to 2.2 mol %, the film growth rate dropped in the case of the high-temperature solutions of batches I–III, containing 0.03 mol % CeO₂ (indicating that the system left the growth region), and increased in the case of the high-temperature solutions of batches IV–VI, whose growth charge contained 0.2 mol % CeO₂ (Table 1). Figure 1 shows vertical sections through the phase diagram in question, the liquidus curve (curve I), a non-equilibrium solidus curve (curve 2), and the growth temperatures of the films. The saturation temperature was constant in the case of film growth from the high-temperature solutions of batches I–III and increased in the case of growth from batches IV–VI.

As in a previous study [1], two growth regions were identified in the case of film growth from the high-temperature solutions of batches I–III. In one region, with a relatively low supercooling ($\Delta t < 25^{\circ}$ C), we obtained pale yellow films. In the other region, with large supercooling ($\Delta t > 25^{\circ}$ C), pale violet films were obtained. All of the films grown from the high-temperature solutions of batches IV–VI were yellowish-green in color.

At certain supercoolings, the LPE growth of the epitaxial films was accompanied by spontaneous crystal growth on the surface of the high-temperature solution, the walls of the platinum crucible, and the surface of the platinum holder. The resultant garnet crystals were tetragon-trioctahedral in shape and had {211} faces, like in Vasil'eva et al. [1], with the tetragon

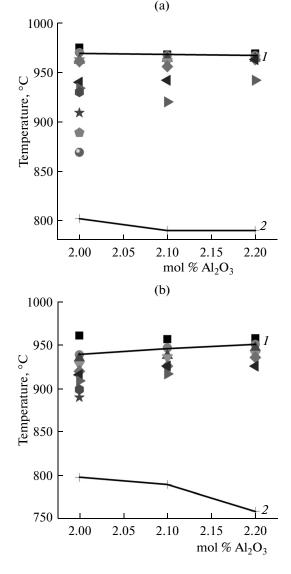
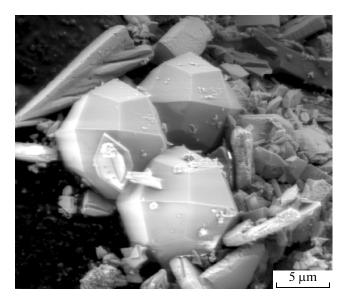


Fig. 1. Vertical sections through the phase diagram of the pseudoternary system $Gd_2O_3-CeO_2-Ga_2O_3-Al_2O_3-(PbO+B_2O_3)$ at (a) 0.03 and (b) 0.2 mol % CeO_2 ; (1) liquidus curve, (2) nonequilibrium solidus curve.

edges no less than 4 and 2.5 μ m in length (Fig. 2). The crystals shown in Fig. 2 grew for 2 h, together with film II-1 (Table 2). The Pb ion concentration in the crystals was found to be about 20 times that in the film.

The sum thickness (2h) of the films grown on the two sides of a substrate was determined gravimetrically [7]. The chemical composition of the films was determined using an FEI Quanta 3D field emission gun, focused ion beam scanning electron microscope. The films were characterized by X-ray diffraction on a Bruker D8 Discover A25 Da Vinsi Design X-ray diffractometer (CuK_{α} radiation). Transmission spectra of the films were measured on a PerkinElmer Lambda 900 spectrophotometer at room temperature in the wavelength range from 186 to 860 nm. Normalized



 $\begin{array}{lll} \textbf{Fig. 2.} & \text{Micrograph of garnet crystals with the compositions} & Pb_{0.40}Ce_{0.03}Gd_{2.57}Al_{2.4}Ga_{2.60}O_{12} & \text{(left),} \\ Pb_{0.49}Ce_{0.03}Gd_{2.48}Al_{2.65}Ga_{2.35}O_{12} & \text{(top right),} & \text{and} \\ Pb_{0.44}Ce_{0.05}Gd_{2.51}Al_{1.93}Ga_{3.07}O_{12} & \text{(bottom right)} \\ \text{spontaneously nucleated on the surface of the high-temperature solution during the growth of a} \\ Pb_{0.02}Ce_{0.02}Gd_{2.96}Al_{2.17}Ga_{2.83}O_{12} & \text{epitaxial film (II-1).} \end{array}$

optical density (D/2h) spectra were calculated from the transmission spectra using the relation D =[ln($T_{\rm sab}/T_{\rm sam}$)], where $T_{\rm sab}$ is the transmission spectrum of the substrate and $T_{\rm sam}$ is the transmission spectrum of the sample. Luminescence and luminescence excitation spectra were measured in the visible and ultraviolet spectral regions using a spectroscopic system built around an LOT-Oriel MS-257 spectrograph. We measured luminescence spectra in the range 350— 850 nm and luminescence excitation spectra in the range 300-500 nm at room temperature. When the luminescence spectra were measured at an excitation wavelength of 440 nm, an OS-11 filter, absorbing light at wavelengths shorter than 510 nm, was placed in front of the entrance slit of the spectrograph. The luminescence spectra were normalized to the instrumental function of the spectroscopic system.

RESULTS AND DISCUSSION

Ce³⁺ luminescence was observed in all of the films grown from the six batches of high-temperature solutions. To compare luminescence spectra, the film with the highest luminescence intensity was chosen from each batch (Table 2).

The luminescence spectra of the films are presented in Figs. 3 and 4. The spectra are dominated by a broad, composite band centered at 550 nm, due to the Ce^{3+} $5d-^2F_{7/2,5/2}$ radiative transitions. With increasing Al concentration, the Ce^{3+} luminescence intensity in the films grown from the high-temperature solutions containing 0.03 mol % CeO_2 increases by a

Table 2. Growth characteristics of the Gd₃(Al,Ga)₅O₁₂:Ce epitaxial films

Film	mol % Al ₂ O ₃	mol % CeO ₂	Film composition	t _g ,°C	Δt,°C	h, μm	λ_{Ce} , nm	
			1 mil composition				5 <i>d</i> ₁	5d ₂
I-1	2.0	0.03	$Pb_{0.02}Ce_{0.02}Gd_{2.96}Al_{2.13}Ga_{2.87}O_{12}$	962	8	15.9	435.8	342.5
II-1	2.1	0.03	$Pb_{0.02}Ce_{0.02}Gd_{2.96}Al_{2.17}Ga_{2.83}O_{12}$	964	3	30.4	437	340.9
III-1	2.2	0.03	$Pb_{0.01}Ce_{0.02}Gd_{2.97}Al_{2.02}Ga_{2.98}O_{12}$	963	4	17.0	434.7	325.9
IV-1	2.0	0.2	$Pb_{0.02}Ce_{0.06}Gd_{2.93}Al_{2.09}Ga_{2.91}O_{12}$	934	4	17.0	437.9	341.9
V-1	2.1	0.2	$Pb_{0.04}Ce_{0.07}Gd_{2.89}Al_{2.13}Ga_{2.87}O_{12}$	925	21	20.9	438.2	341.7
VI-1	2.2	0.2	$Pb_{0.02}Ce_{0.06}Gd_{2.92}Al_{2.22}Ga_{2.78}O_{12}$	925	25	18.2	438.6	340.4

 $t_{\rm g}$ is the growth temperature and Δt is supercooling.

factor of 2.5 under excitation at 340 nm and by a factor of 3 at an excitation wavelength of 440 nm. The highest luminescence intensity under excitation at 340 (Fig. 3a) and 440 nm (Fig. 4a) is offered by the $Pb_{0.02}Ce_{0.02}Gd_{2.96}Al_{2.17}Ga_{2.83}O_{12}$ film (II-1), which has the highest Al ion concentration (x = 2.17) and contains 0.7 at % Ce^{3+} . At wavelengths from 350 to 450 nm, the luminescence spectra of films III-1 and II-1 presented in Fig. 3a show additional luminescence bands, which correspond to the luminescence of Tb^{3+} ions in low concentration [8]. It seems likely that this emission arises from Tb^{3+} ions in the GGG substrate, whose spectrum is shown in Fig. 3a (spectrum I). The excitation is only partially absorbed by the Ce^{3+} ions because of their low concentration.

In the films grown from the high-temperature solutions containing 0.2 mol % CeO_2 , no emission from Tb^{3+} was detected, because excitation was completely absorbed by the Ce^{3+} ions owing to their increased concentration (Fig. 3b). With increasing Al concentration, the Ce^{3+} luminescence intensity in these films increases five times under excitation at 340 nm (Fig. 3b) and three times at an excitation wavelength of 440 nm

(Fig. 4b). The $Pb_{0.02}Ce_{0.06}Gd_{2.92}Al_{2.22}Ga_{2.78}O_{12}$ film (VI-1), which has x=2.22 and contains 2 at % Ce^{3+} , shows the highest luminescence intensity under excitation at 340 and 440 nm. According to our experiments, the intensity of the luminescence bands of the $Pb_{0.02}Ce_{0.06}Gd_{2.92}Al_{2.22}Ga_{2.78}O_{12}$ film (VI-1) is higher than the luminescence intensity of the $Pb_{0.02}Ce_{0.02}Gd_{2.96}Al_{2.17}Ga_{2.83}O_{12}$ film (II-1) by a factor of 1.2 under excitation at 340 nm and by a factor of 1.5 at an excitation wavelength of 440 nm. The rise in luminescence intensity is due to the increase in Al content from x=2.17 to 2.22 and the increase in Ce^{3+} content from 0.7 to 2 at %.

Figure 5 shows spectral dependences of the normalized optical density of the films. According to Vasil'eva et al. [7], the absorption band at a wavelength of 276 nm (36 230 cm⁻¹) corresponds to the ${}^{1}S_{0} \rightarrow {}^{3}P_{1}$ electronic transition of the Pb²⁺ ion (6s²) {Pb²⁺ (6s², ${}^{1}S_{0}) \rightarrow$ Pb²⁺ (6s¹ 6p¹, ${}^{3}P_{1}$)}. The two broad absorption bands correspond to the 4f (${}^{2}F_{5/2,7/2}$)–5d electronic transition of the Ce³⁺ ion. In the case of films I-1 and II-1, grown from the high-temperature solutions containing 2.0 and 2.1 mol % Al₂O₃, respectively, the

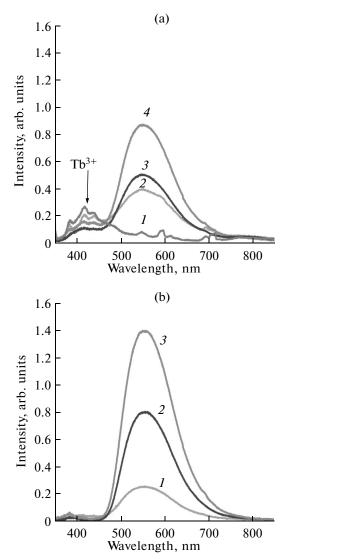
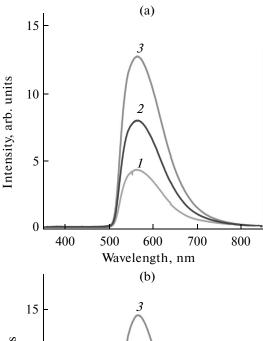


Fig. 3. Luminescence spectra of (a) a GGG substrate (I); films grown from the high-temperature solutions containing 0.03 mol % CeO₂, namely, films III-1 (2), I-1 (3), and II-1 (4), and (b) films grown from the high-temperature solutions containing 0.2 mol % CeO₂, namely, films IV-1 (I), V-1 (2), and VI-1 (3) (see Table 2); excitation at 340 nm.

absorption band of the $5d_1$ level shifts from 435.8 (22 950 cm⁻¹) to 437 nm (22 880 cm⁻¹), that is, by 1.2 nm to longer wavelengths, and the band of the $5d_2$ level shifts from 342.5 (29 200 cm⁻¹) to 340.9 nm (29 330 cm⁻¹), that is, by 1.6 nm to shorter wavelengths, as the Al ion content increases from x = 2.13 to 2.17 (Fig. 5a). Film III-1, grown from the high-temperature solution containing 2.2 mol % Al₂O₃, has x = 2.06, which is considerably lower than the Al content of films I-1 and II-1. Because of this, the absorption band of the $5d_1$ level in the spectral dependence of the normalized optical density for this film is shifted to shorter wavelengths and that of the $5d_2$ level is shifted



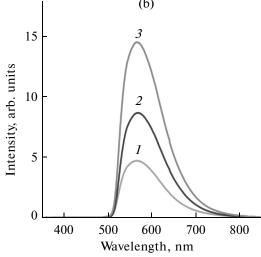


Fig. 4. Luminescence spectra of (a) epitaxial films grown from the high-temperature solutions containing 0.03 mol % CeO_2 , namely, films III-1 (1), I-1 (2), and II-1 (3), and (b) films grown from the high-temperature solutions containing 0.2 mol % CeO_2 , namely, films IV-1 (1), V-1 (2), and VI-1 (3) (see Table 2); excitation at 440 nm.

to longer wavelengths with respect to the corresponding absorption bands of films I-1 and II-1.

Increasing the CeO₂ content of the growth charge from 0.03 to 0.2 mol % increases the intensity of the absorption band of the Ce³⁺ $5d_1$ level in the films by four times. In films IV-1, V-1, and VI-1, grown from the high-temperature solutions containing 2.0, 2.1, and 2.2 mol % Al₂O₃, the absorption bands of the $5d_1$ level shift to longer wavelengths from 437.9 (22 840 cm⁻¹) to 438.6 nm (22 800 cm⁻¹) (by 0.7 nm) and those of the $5d_2$ level shift to shorter wavelengths from 341.9 (29 250 cm⁻¹) to 340.4 nm (29 380 cm⁻¹) (by 1.5 nm) as x increases from 2.09 to 2.22 (Fig. 5b).

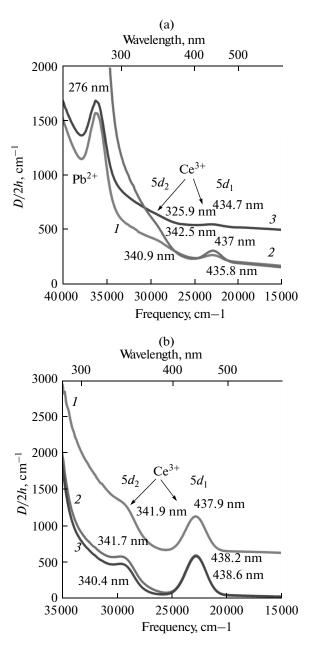


Fig. 5. Spectral dependences of the normalized optical density D/2h vs. frequency for epitaxial films grown from the high-temperature solutions containing (a) 0.03 and (b) 0.2 mol % CeO₂: (a) (*I*) I-1, (*2*) II-1, (*3*) III-1; (b) (*I*) IV-1, (*2*) V-1, (*3*) VI-1 (see Table 2).

Figure 6 shows the excitation spectra of the luminescence at a wavelength of 530 nm for films IV-1, V-1, and VI-1. The spectra are normalized to the intensity of the band centered at 450 nm. There are two bands corresponding to f-d transitions of the Ce³⁺ ion.

Figure 7 illustrates the difference in surface morphology between films II-1 and VI-1, which were grown from high-temperature solutions using growth charges containing 0.03 and 0.2 mol % and showed the highest luminescence intensity. It is seen that the films have rough surfaces, consisting of intergrowths of sin-

gle-crystalline grains with an edge length no greater than 6 (Fig. 7a) or 4 μ m (Fig. 7b), oriented both in the direction of the [111] axis and in the plane of the substrate. X-ray diffraction patterns obtained in the $\theta/2\theta$ scan mode showed only strong 444 and 888 reflections from film VI-1 and weak 444 and 888 reflections from its substrate, shielded by the film (Fig. 8). From the peak positions, we determined the unit-cell parameters of the substrate, a_s = 12.3829 Å, and film, a_f = 12.269 Å (the relative lattice mismatch ($\Delta a_s - a_f$)/ a_f is 0.9%). The unit-cell parameter of the substrate agrees with JCPDS Powder Diffraction File data for GGG

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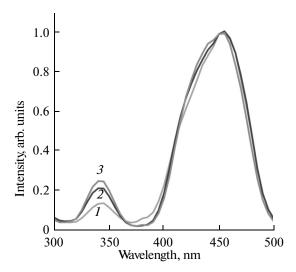
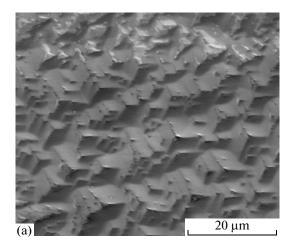
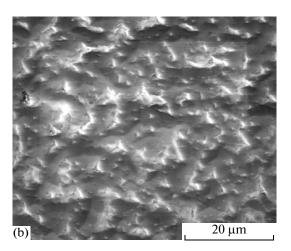
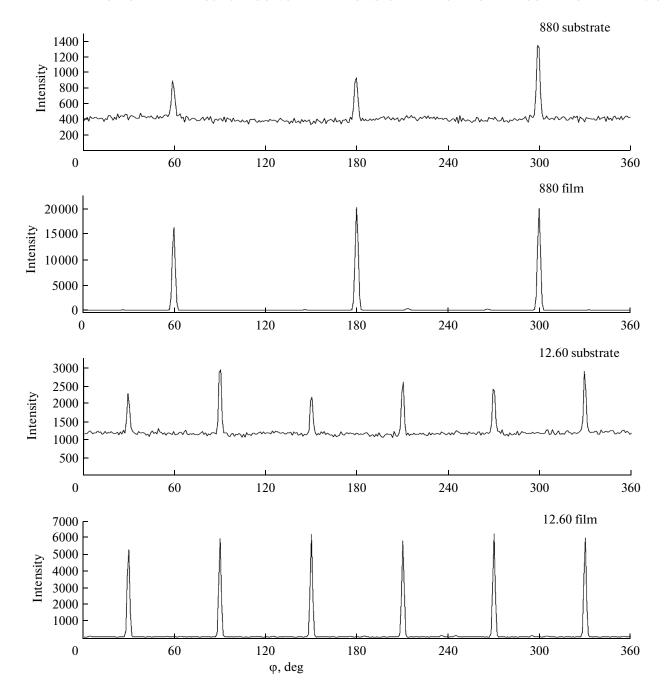


Fig. 6. Excitation spectra of the luminescence at a wavelength of 530 nm for epitaxial films grown from high-temperature solutions using growth charges containing 0.2 mol % CeO_2 : (1) IV-1, (2) V-1, (3) VI-1 (see Table 2).







 $\begin{tabular}{lll} Fig. 8. A symmetric 880 and 12.60 reflections obtained via azimuthal scanning of the GGG substrate and $Pb_{0.02}Ce_{0.06}Gd_{2.92}Al_{2.22}Ga_{2.78}O_{12} (VI-1) film. \end{tabular}$

(card no. 76-2290). The presence of only one type of reflection suggests that the films either are single-crystalline or have a perfect axial texture. The profiles of the asymmetric 880 and 12.60 reflections in Fig. 8 indicate that not only [111] but also all of the other crystallographic directions in film VI-1 and its GGG substrate coincide. Thus, film VI-1 grew epitaxially on its substrate and was single-crystalline.

CONCLUSIONS

Twenty-five films have been grown on (111)-oriented GGG substrates by LPE from high-temperature solutions using solvents of the PbO $-B_2O_3$ system and growth charges containing 2.0, 2.1, or 2.2 mol % Al_2O_3 and 0.03 or 0.2 mol % CeO_2 . The spectral dependences of the normalized optical density of the films showed two broad absorption bands of the Ce^{3+} ion,

corresponding to the $4f(^2F_{5/2,7/2})-5d$ electronic transition, and an absorption band of the Pb²⁺ impurity ion, centered at a wavelength of 276 nm and corresponding to the $^1S_0 \rightarrow {}^3P_1$ electronic transition. With increasing Al concentration in the films, the absorption bands of the Ce³⁺ $5d_1$ level shifted to longer wavelengths and those of the $5d_2$ level shifted to shorter wavelengths. The highest Ce³⁺ luminescence intensity was observed in the Pb_{0.02}Ce_{0.06}Gd_{2.92}Al_{2.22}Ga_{2.78}O₁₂ film, which had x = 2.22 and contained 2 at % Ce³⁺.

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