Luminescent Properties of Cr-Doped Gallium Garnet Crystals Grown by the Micro-Pulling-Down Method

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Abstract

Cr-doped $(\text{Gd}_x \text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ crystals ($x = 0.00, 0.25, 0.50, 0.75$ and 1.00) were grown by the micro-pulling-down method and examined for their possible application as red and infrared scintillating detectors in medical field. Although $\text{Cr}:(\text{Gd}_{0.75}\text{Y}_{0.25})_3\text{Ga}_5\text{O}_{12}$ and $\text{Cr}:\text{Gd}_3\text{Ga}_5\text{O}_{12}$ had similar X-ray diffraction patterns, other samples showed some change in lattice constant. All the crystals had broad emission bands in the red and infrared region when excited by either 450 nm photons or X rays. These bands were associated with ${}^4\text{T}_2 \rightarrow \text{A}_2$ transitions. Moreover, redshift of the emission-peak wavelengths (${}^4\text{T}_2 \rightarrow \text{A}_2$) and absorption peaks ($\text{A}_2 \rightarrow {}^4\text{T}_1$ and ${}^4\text{T}_2$) was observed with increase of Gd content (x) in Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ due to the change of the crystal fields. The crystals had scintillation emissions in the wavelength region suitable for the real time dose monitoring in radiation therapy.

Key words:

A2: Single crystal growth

B1: Gallium compounds

B2: Scintillator materials

B3: Scintillators

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1. Introduction

Scintillators are widely used in medical imaging, astronomy, oil well logging, and other applications to measure radiation (X/gamma-ray energy, count rate, etc.) in real time [1]. On the other hand, some dosimeters are readout after the irradiation. In radiation therapies, X-rays, gamma rays, protons, and neutrons are used for the tumor treatments. In such cases, accurate control of intensity of the treatment is desired to reduce or avoid unwanted side effects. However, it is difficult to determine the precise dose in real time. On the other hand, overdose or incorrect positioning of the human tissue during the irradiation may lead to medical accidents [2].

Infrared scintillator can be used for the real-time dose monitoring in such therapies, because infrared photons can pass through the human tissues. The infrared light emitted from an infrared scintillator, injected via a catheter into a tumor, can be detected using a charge-coupled device or complementary metal oxide semiconductor camera placed outside the patient's body. This way, the intensity of the infrared light can be associated with the dose received by the patient during the therapy. This feedback allows immediate control of the radiation intensity, and it is anticipated that application of such system may reduce the number of medical accidents [3]. This is especially important for the X-ray therapy, because even a scintillating sensor of small size is sufficient to absorb X-ray photons.

The infrared scintillators applied to such real time monitoring must satisfy three conditions as follows. First of all, the emission wavelength should correspond to the low absorption region for human tissue, that is 650-1200 nm. Secondly, the X-ray stopping power should be sufficiently high for good detection efficiency for a small size scintillator (few mm in diameter) suitable for implantation. Finally, the scintillating material must be non-hygroscopic and non-toxic.

In the past, Nakata et al. used Cr-doped Al_2O_3 ceramics [3]. However, their light output was low. Up to now, we have investigated the scintillation properties of Nd: Lu_2O_3 and Cr:(Gd,Y) $_3\text{Al}_5\text{O}_{12}$ [4, 5]; Nd-doped samples had emission lines at over 950 nm originating from 4f-4f transition of Nd^{3+} , however, photo

detectors have low detection efficiency in this wavelength region compared with the typical emission region of the d-d transitions of Cr^{3+} such as ${}^4\text{T}_1 \rightarrow \text{A}_2$, ${}^4\text{T}_2 \rightarrow \text{A}_2$ or ${}^2\text{E} \rightarrow {}^4\text{A}_2$ transitions. Here, It is known that light output is in inverse proportion to bandgap energy, and the $\text{Y}_3\text{Ga}_5\text{O}_{12}$ (YGG) crystal has smaller band gap energy (E_{gap}) compared with $\text{Y}_3\text{Al}_3\text{O}_{12}$ (YAG), ($E_{\text{gap}} = 6.5$ and 5.5 eV for YAG and YGG, respectively) [6]. Thus, Cr:YGG is expected to have higher light output than Cr:YAG. Moreover, $\text{Gd}_5\text{Al}_3\text{O}_{12}$ is an incongruently melting compound, while the crystals of $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$) series melt congruently. The purpose of this report was to discuss melt growth and scintillation properties of Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$) crystals as alternative candidates for red/infrared scintillator applications.

2. Materials and Experimental Methods

Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$) crystals were grown by the micro-pulling-down (μ -PD) method [7, 8]. The nominal melt compositions are listed in Table 1. The starting materials were 99.99%-purity Cr_2O_3 , Gd_2O_3 , $\beta\text{-Ga}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$ powders. The growths were performed from iridium crucible with constant pulling rate of 0.05 mm/min in nitrogen atmosphere (flow).

Parts of the grown crystals were crushed into powders, and the powder X-ray diffraction (XRD) analysis was carried out in the 2θ range of $15\text{-}80^\circ$ using a diffractometer (RIGAKU, RINT 2000); the X-rays were generated from a Cu-K α target with accelerated electrons (accelerating voltage of 40 kV and beam current of 40 mA). The crystalline structures were identified by comparison of the obtained XRD patterns and corresponding patterns available from JCPDS database. Each as-grown crystal was sliced and mirror-polished for inspection of its optical properties. The thicknesses of the specimens were 1 mm. Actual chemical compositions on the surface along sliced samples (cross section samples cut perpendicular to the growth axis) were determined using Electron Probe Microanalysis (EPMA) (JEOL, JXA-8621MX). These measurements were performed at voltage of 20 kV and current of 1.2×10^{-8} A.

Transmittance spectra were examined with a spectrophotometer (JASCO: V-550) in the wavelength range from 200 to 900 nm. Photo-luminescence spectra of the samples were also inspected with a

photo-luminescence measurement system (Hamamatsu: C9920-03G). The excitation light was generated by a Xe lamp (Hamamatsu: L9799-02), and all the excitation wavelengths were approximately 450 nm. The radio-luminescence spectra under X-ray excitation were recorded with a spectrometer (Andor: DU420-OE) using the same X-ray generator and the conditions applied for the XRD analysis (RIGAKU: RINT-2000).

Table 1: List of the grown crystals of $\text{Cr:}(\text{Gd}_x \text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$)

X	Nominal composition	Abbreviation
x = 0.00	$\text{Y}_3(\text{Ga}_{0.995}\text{Cr}_{0.005})_5\text{O}_{12}$	Cr:YGG
x = 0.25	$(\text{Gd}_{0.25}\text{Y}_{0.75})_3(\text{Ga}_{0.995}\text{Cr}_{0.005})_5\text{O}_{12}$	Cr:Gd25YGG
x = 0.50	$(\text{Gd}_{0.50}\text{Y}_{0.50})_3(\text{Ga}_{0.995}\text{Cr}_{0.005})_5\text{O}_{12}$	Cr:Gd50YGG
x = 0.75	$(\text{Gd}_{0.75}\text{Y}_{0.25})_3(\text{Ga}_{0.995}\text{Cr}_{0.005})_5\text{O}_{12}$	Cr:Gd75YGG
x = 1.00	$\text{Gd}_3(\text{Ga}_{0.995}\text{Cr}_{0.005})_5\text{O}_{12}$	Cr:GGG

3. Results and discussion

Fig.1 illustrates the visual appearance of the grown crystals. The solidification fractions were 84, 69, 71, 82 and 80% for the Cr:YGG, Cr:Gd25YGG, Cr:Gd50YGG, Cr:Gd75YGG and Cr:GGG melts, respectively. These values are sufficiently high, proving that the melts used were not very distant from congruently melting compositions. Figure 2 shows the powder X-ray diffraction patterns of these samples. All the measured diffraction patterns agreed well with those corresponding to $\text{Y}_3\text{Ga}_5\text{O}_{12}$ (JCPDS No. 43-0512, cubic crystal, Space group: $\text{Ia}\bar{3}\text{d}$) or $\text{Gd}_3\text{Ga}_5\text{O}_{12}$ (JCPDS No. 13-0493, cubic crystal, Space group: $\text{Ia}\bar{3}\text{d}$). Moreover, the peaks detected for the Cr:GGG crystal were slightly shifted to the low angle side compared with Cr:YGG. Since Gd^{3+} and Y^{3+} ion radii in the dodecahedral site are 1.05-1.06 and 1.019 Å, respectively [9,10], this shift is reasonable [9, 10]. Meanwhile, Cr:GGG and Cr:Gd75YGG had almost the same pattern,

and both lattice constants appeared to be nearly the same as listed in Table 2.

Table 2: Lattice constants (a) of the samples

Lattice constants (a) [$\text{\AA}$]	
Cr:YGG	12.27 ± 0.01
Cr:Gd25YGG	12.34 ± 0.01
Cr:Gd50YGG	12.40 ± 0.01
Cr:Gd75YGG	12.40 ± 0.02
Cr:GGG	12.39 ± 0.02

Figure 3 presents the photographs of sliced samples (cross section samples cut perpendicular to the growth axis / pulling axis). Segregation and non-uniform distribution of Cr could be seen for all the samples with naked eyes: the central part of each sample had darker green color noticeably different from that of the peripheral parts (edges). Spatial radial distributions of the observed Cr content (C) divided by the nominal (initial) composition (C_0), C/C_0 , measured for these samples by EPMA are illustrated in Fig. 4.

The Ir crucible used in the μ -PD method growths had a single hole outlet positioned in the geometrical center of the nozzle die, and most of the Cr atoms were accumulated in the part of the crystal matching the position of the outlet as it is seen in Fig 4, a finding which agrees with the >1 values of the distribution segregation coefficient of Cr suggested in [11-13]

The other cations, Gd^{3+} , Y^{3+} and Ga^{3+} were distributed uniformly on the surfaces of all samples, and

their average C/C_0 values are listed in Table 3.

Table 3: C/C_0 values for $\text{Cr:}(\text{Gd}_x \text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$) based on EPMA measurements

Nominal composition		C/C_0^*		
		Gd	Y	Ga
Cr:YGG	$\text{Y}_3(\text{Ga}_{0.995} \text{Cr}_{0.005})_5\text{O}_{12}$	-	1.13 ± 0.03	0.92 ± 0.02
Cr:Gd25YGG	$(\text{Gd}_{0.25} \text{Y}_{0.75})_3(\text{Ga}_{0.995} \text{Cr}_{0.005})_5\text{O}_{12}$	1.06 ± 0.07	1.06 ± 0.05	0.95 ± 0.02
Cr:Gd50YGG	$(\text{Gd}_{0.50} \text{Y}_{0.50})_3(\text{Ga}_{0.995} \text{Cr}_{0.005})_5\text{O}_{12}$	1.10 ± 0.08	1.03 ± 0.09	0.95 ± 0.01
Cr:Gd75YGG	$(\text{Gd}_{0.75} \text{Y}_{0.25})_3(\text{Ga}_{0.995} \text{Cr}_{0.005})_5\text{O}_{12}$	1.08 ± 0.04	1.05 ± 0.08	0.94 ± 0.01
Cr:GGG	$\text{Gd}_3(\text{Ga}_{0.995} \text{Cr}_{0.005})_5\text{O}_{12}$	1.09 ± 0.03	---	0.94 ± 0.02

* Value = (average $\pm$ standard deviation)

The transmittance spectra of the crystals are shown in Fig. 5. All the spectra had two broad absorption-bands centered around 450 and 620 nm wavelengths. These bands originated from d-d transitions of Cr^{3+} : from the ground state (4A_2) to its excited states 4T_1 and 4T_2 for the 450- and 620-nm bands, respectively. In addition, two other absorption peaks associated with Gd^{3+} were also detected in the vicinity of 310 nm wavelength, and they were ascribed to 4f – 4f transitions ($^8S_{7/2} \rightarrow ^6P_{7/2-3/2}$ of Gd^{3+}) [14]. Since lattice constants and crystal fields changed depending on crystal composition, absorption wavelengths of the d-d transitions were also displaced as it is illustrated in Fig. 6.

In the photo-luminescence spectra, each sample demonstrated broad band associated with $^4T_2 \rightarrow A_2$ transitions, and a detectable narrow peak at approximately 693 nm originating from $^2E \rightarrow ^4A_2$ transition (R-lines) was clearly observed for the Cr:YGG (Fig. 7) [15-17]. Although other samples should also have

these R-lines, such peaks were not well-seen. The difference between Cr:YGG and the other crystals can be understood as follows. Cr:YGG has the smallest lattice constant among the materials discussed here. Therefore, it would have stronger crystal fields, and its excitation states of 2E and 4T_2 are overlapped in Tanabe-Sugano diagram for the d^3 transition metal cations such as Cr^{3+} , V^{2+} and Mn^{4+} . On the other hand, the other samples have lower excitation states of 4T_2 due to weaker crystal fields than the fields of the Cr:YGG [18, 19].

Figure 8 shows radio-luminescence spectra for the samples, and all the samples indicated red-infrared scintillation emission. These spectra illustrate that increase of the Gd^{3+} content in the $Cr:(Gd_xY_{1-x})_3Ga_5O_{12}$ series resulted in red-shift of the emission peak wavelength as a result of change of the crystal fields. However, the Cr:GGG had a peak wavelength similar to that of Cr:Gd75YGG since the lattice constants of these crystals are almost the same according to their X-ray diffraction patterns (Fig. 2). Thus, their crystal fields are almost identical. Compared with photo-luminescence spectra, sharp peaks for the Cr:YGG were not detected in radio-luminescence measurements, because the detector used in the radio-luminescence characterization apparatus had worse wavelength resolution than the photo-luminescence detector. Rough estimation of the relative radio-luminescence outputs was also performed, correcting the detection efficiency as function of wavelength. Figure 9 shows the relative light outputs for the $Cr:(Gd_xY_{1-x})_3Ga_5O_{12}$ specimens ($x = 0.00, 0.25, 0.50, 0.75, 1.00$) in comparison with that of Cr:YAG. It is well seen that the light outputs of all the $Cr:(Gd_xY_{1-x})_3Ga_5O_{12}$ crystals exceed that of Cr:YAG. One of the reasons for the greater light output observed in the $Cr:(Gd_xY_{1-x})_3Ga_5O_{12}$ crystals would be associated with their smaller energy bandgaps as compared with Cr:YAG. On the other hand, some samples ($x = 0.25, 0.50$ and 0.75) had lower light outputs than those of Cr:YGG and Cr:GGG due to reduced efficiency of energy transportation to emission centers by some traps.

4. Conclusions

Cr-doped $(Gd_xY_{1-x})_3Ga_5O_{12}$ crystals ($x = 0.00, 0.25, 0.50, 0.75$, and 1.00) were grown by the

micro-pulling-down method for applications as red and infrared scintillators for medical therapy. All the crystals had broad emission bands in the red and infrared regions when excited by either 450 nm photons or X-rays. Formation of these bands was associated with ${}^4T_2 \rightarrow A_2$ transitions. Moreover, redshifts of the emission peaks' wavelengths were observed when the Gd^{3+} content in the Cr-doped $(Gd_xY_{1-x})_3Ga_5O_{12}$ crystals increased due to the change of the crystal fields. All scintillation emission peaks of these crystals were positioned in the wavelength regions suitable for actual medical applications.

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Table captions

Table 1. List of the grown crystals of $\text{Cr:}(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$)

Table 2. C/C_0 values for $\text{Cr:}(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ ($0 \leq x \leq 1$) based on EPMA measurements

Figure captions

Figure1. View of the as-grown Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ crystals, where $x = 0.00$ (a), 0.25 (b), 0.50 (c), 0.75 (d) and 1.00 (e). (f) shows schematic view of micro-pulling down method and the definition of the pulling axis.

Figure2. (i) X-ray diffraction patterns for Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ crystals, where $x = 0.00$ (a), 0.25 (b), 0.50 (c), 0.75 (d) and 1.00 (e), and (ii) fragment of the XRD results for 2θ ranged from 28 to 36° for the same crystals.

Figure3. View of the polished Cr-doped $(\text{Gd}_x\text{Y}_{1-x})_3\text{Ga}_5\text{O}_{12}$ samples cut perpendicular to the growth axis, where $x = 0.00$ (a), 0.25 (b), 0.50 (c), 0.75 (d) and 1.00 (e).

Figure4. (a) Schematic view of the scanning direction (arrow) for the SEM/EPMA characterization on the surface of the (Cr:YGG) sample (Fig.3), and EPMA results on Cr distribution for the Cr-doped

($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ samples, where $x = 0.00$ (b), 0.25 (c), 0.50 (d), 0.75 (e) and 1.00 (f).

Figure5. Transmittance spectra of the Cr-doped ($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ crystals, where $x = 0.00$ (narrow solid line), 0.25 (gray- dashed line), 0.50 (gray- solid line), 0.75 (black-dashed line) and 1.00 (black-thick-solid line).

Figure6. Peak wavelength of absorption band for $^4\text{A}_2$ to $^4\text{T}_1$ (a) and $^4\text{T}_2$ (b) as a function of x index in the Cr-doped ($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ crystals.

Figure7. Photo-luminescence spectra of the Cr-doped ($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ crystals for $x = 0.00$ (narrow solid line), 0.25 (gray- dashed line), 0.50 (gray- solid line), 0.75 (black-dashed line) and 1.00 (black-thick-solid line) excited by 450 nm photons.

Figure8. Radio-luminescence spectra of the Cr-doped ($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ crystals for $x = 0.00$ (narrow solid line), 0.25 (gray- dashed line), 0.50 (gray- solid line), 0.75 (black-dashed line) and 1.00 (black-thick-solid line) excited by X-rays.

Figure9. Relative light outputs normalized to that of the Cr:YAG for the Cr-doped ($\text{Gd}_x\text{Y}_{1-x}$) $_3\text{Ga}_5\text{O}_{12}$ samples.

Figure1

Figure2

(a) Cr:YGG

(b) Cr:Gd25YGG

(c) Cr:Gd50YGG

(d) Cr:Gd75YGG

(e) Cr:GGG

10 mm
↔

Figure4

(a) Cr:YGG

Figure5

Figure6

Figure7

Figure8

Figure9