

ลักษณะเฉพาะด้านลูมิเนสเซนส์ของแก้วซิลิกาเจือด้วยอิตอน Ce^{3+} เมื่อกระตุ้นด้วยรังสีอัลตราไวโอเล็ตและรังสีเอกซ์

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บทคัดย่อ

แก้วซิลิกาเจือด้วยอิตอน Ce^{3+} ถูกเตรียมขึ้นด้วยวิธีการแทรกซึมของสารละลายซีเรียมไนเตรต [$Ce(NO_3)_3$] เข้าไปในแก้วซิลิกาพอร์ตามด้วยการอบที่อุณหภูมิสูงในบรรยากาศของก๊าซคาร์บอนมอนนอกไซด์ (CO) เมื่อกระตุ้นด้วยรังสีอัลตราไวโอเล็ตและรังสีเอกซ์ พบว่าแก้วซิลิกาเจือด้วยอิตอน Ce^{3+} จะปลดปล่อยสเปกตรัมแสงลูมิเนสเซนส์ออกมา โดยมีความเข้มสูงสุดอยู่ที่ความยาวคลื่น 375 nm ซึ่งเกิดจากการทรานซิชันของระดับพลังงาน 5d ไปสู่ระดับพลังงาน 4f ของอิตอน Ce^{3+} เมื่อกระตุ้นด้วยรังสีอัลตราไวโอเล็ต พบว่าแสงลูมิเนสเซนส์ที่ปลดปล่อยออกมาสลายตัวด้วยค่าคงที่การสลายตัวประมาณ 50-70 ns เมื่อกระตุ้นด้วยรังสีเอกซ์จากหลอดเอกซเรย์ พบว่าแก้วซิลิกาเจือด้วยอิตอน Ce^{3+} ให้ค่าประสิทธิภาพซินทิลเลชันรวมที่สูงเทียบเคียงได้กับผลึกบิสมีทเจอร์มาเนต $Bi_4Ge_3O_{12}$ (BGO) และให้ค่าyieldแสงประมาณ 16% ของ yieldแสงที่ได้จากผลึก BGO เมื่อกระตุ้นด้วยรังสีแกมมา

คำสำคัญ : Ce^{3+} / yieldแสง / ลูมิเนสเซนส์ / การสลายตัวของแสงลูมิเนสเซนส์ / แก้วซิลิกา / ซินทิลเลชัน

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Luminescence Characteristics of Ce³⁺- Doped Silica Glass under UV and X-ray Excitation

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Abstract

Ce³⁺- doped silica glass was prepared by impregnation of Ce ions into a porous silica glass followed by high temperature sintering in a CO reducing atmosphere. The characteristic emission of Ce³⁺ 5d → 4f transition peaking around 375 nm is observed in the luminescence spectra under UV and X-ray excitation. The photoluminescence decay is governed by a few tens of nanoseconds decay time. The integral scintillation efficiency under X-ray irradiation is comparable to that of a Bi₄Ge₃O₁₂ (BGO) crystal while the light yield of about 16% of that of the BGO is obtained under γ -ray excitation.

Keywords : Ce³⁺ / Light yield / Luminescence / Luminescence decays / Silica glass / Scintillation

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1. Introduction

Silica glass is an attractive host material used for studying the luminescent properties of rare earth and transition metal ions because it has good optical and mechanical properties as well as good chemical durability. Scintillating glasses can be used for the detection of X-rays, γ -rays and neutrons [1–3]. They are cheaper and easier to fabricate with respect to single crystal materials, but they suffer from inefficient energy transfer and concentration quenching resulting from the interaction of emission activator ions. The concentration quenching is closely related to the phenomenon of phase separation, which occurs mainly during glass melting [4]. Solubility of rare-earth (RE) ions is low in silica glass prepared by direct high-temperature melting of RE compounds and raw materials of glass, and easily leads to segregation or phase separation even at low concentration.

In order to suppress concentration quenching of emission ions to obtain intense luminescence glass, our group have developed a novel method based on fabrication and sintering technique for uniform distribution of RE ions in high silica glass host [5-7]. In contrast to the direct introduction of RE ions into silica glasses by high-temperature melting, a three-step process was employed including preparation of a porous silica glass, adsorption of RE ions and high-temperature sintering in a reducing atmosphere. With very large specific surface area and uniform distribution of the pore channels of the obtained porous glass, the RE dopants can be adsorbed and distributed uniformly in the glass host. Very intense photoluminescence emissions were observed in the leached, sintered high silica glasses impregnated with Tb^{3+} [8,9] and Eu^{2+} [3,10,11] ions under UV excitation. It showed that this novel method can uniformly distribute

the RE dopants in the glass host without clusters occurring in the form of RE-O-RE bonds. It confirmed the success in solving the mentioned problem of concentration quenching of emission ions.

The $5d \rightarrow 4f$ emission of Ce^{3+} occurs usually at shorter wavelength range than that of Eu^{2+} when both ions are at same host lattice site. Moreover, the fully allowed fluorescence transition of Ce^{3+} is faster than that of Eu^{2+} . This feature makes the Ce^{3+} -doped inorganic materials good candidates for fast scintillators [12]. In this paper, we prepared Ce^{3+} -doped silica glass by impregnation of Ce ions into a porous silica glass followed by high-temperature sintering in a CO reducing atmosphere. The luminescence characteristics were investigated under UV and X-ray excitation. In order to evaluate its potential in scintillation applications, the integral scintillation efficiency and scintillation light yield (LY) were also determined with a $Bi_4Ge_3O_{12}$ (BGO) scintillation crystal as a reference.

2. Experimental

The porous glass was obtained by leaching the alkaliborate phase from phase-separated alkali-borosilicate glass in hot acid solution [5-7]. Reagent-grade chemicals SiO_2 , H_3BO_3 , Na_2CO_3 , $CaCO_3$, and $Al(OH)_3$ were used as starting materials in a solid-state reaction to produce an initial glass. The initial glass with the composition (in wt %) $51.8SiO_2 \cdot 33.3B_2O_3 \cdot 2.6Al_2O_3 \cdot 4.0CaO \cdot 8.3Na_2O$ was melted in a platinum crucible at $1400^\circ C$ for 4 h. The melt was then poured onto a preheated stainless steel plate and pressed to a thickness of about 2 mm by another plate. Phase separation of alkaliborate and silicate phases was performed at $590^\circ C$ for 40 h. The obtained phase-separated glass was cut to pieces and polished with size of about $8 \times 8 \times 1 \text{ mm}^3$,

followed by leaching in hot (90°C) 1N HNO₃ acid solution for 48 h. After washing with distilled water and drying, leached porous glasses were obtained. The obtained porous glass contains over 96% of SiO₂ [5,6] and is therefore called high silica glass. The pore size was smaller than 12 nm determined by BET technique (Belsorp Mini II, BEL Japan, Inc.) and pores nominally occupied about 40% volume of the glass. They were distributed uniformly in the silica glass [5]. The porous glasses were immersed in the 0.03 M solution of Ce(NO₃)₃ · 6H₂O for 1 h and dried at room temperature (RT) and then sintered at 1100°C in a CO reducing atmosphere for 2 h to obtain nonporous glasses. The obtained Ce glasses are colorless and transparent with the density of 2.2 g/cm³ determined by Archimedes method. The polished plate with size of about 8×8×1 mm³ was used for all the measurements.

Excitation and emission spectra in the UV/visible region were recorded on a Hitachi F-2500 fluorescence spectrophotometer equipped with a 150 W xenon lamp source. Absorption spectrum in the UV/visible region was recorded on a Shimadzu UV-3101PC absorption spectrometer. Radioluminescence (RL) spectra and photoluminescence (PL) decays were obtained using the custom made 5000 M fluorometer, Horiba Jobin Yvon, equipped with X-ray tube (steady-state tube, 40 kV, Mo anode) and pulsed (ns) coaxial H₂ - flashlamp as excitation sources, for details see Ref.[13]. RL spectra were corrected for the wavelength dependence of the photodetector quantum efficiency as well as of the monochromator transmission. The integrated RL spectra measured at the samples of the same shape and same experimental conditions provide a good relative estimate of scintillation efficiency of our

glass sample compared to a BGO crystal. LY measurements were performed under the excitation of ¹³⁷Cs 662 keV gamma rays using a photomultiplier tube (PMT) readout. The signal from the PMT anode was passed to a Canberra 2005 preamplifier and then to a Tennelec TC243 spectroscopy amplifier set at 4 μs shaping time constant. The multichannel analyzer (Tukan 8k MCA) was used to record the pulse height spectra. For a comparison, BGO single crystal grown by Bridgman method at the Shonan Institute of Technology (Fujisawa, Japan) and CeF₃ single crystal grown by Czochralski method at Crytur Ltd (Czech Republic) of the same size with our glass sample were used as reference scintillators. All the measurements were carried out at RT.

3. Results and discussion

3.1 Photoluminescence of Ce³⁺-doped silica glass

Fig.1 presents the absorption (curve a), excitation (curves b and c) and emission (curve d) spectra at RT for the Ce³⁺-doped silica glass sintered in a reducing (CO) atmosphere. In the absorption and excitation spectra, a broad band peaking at approximately 300 nm can be attributed to the absorption due to the 4f → 5d transitions of Ce³⁺ ions. The higher lying absorption band with an absorption edge around 240 nm is not reflected in the excitation spectrum for the Ce³⁺ emission, so that it might be related to the glass-host absorption. Alternatively, the higher lying 5d states of Ce³⁺ might be situated in the host conduction band and the excitation into them leads rather to Ce³⁺ ionization.

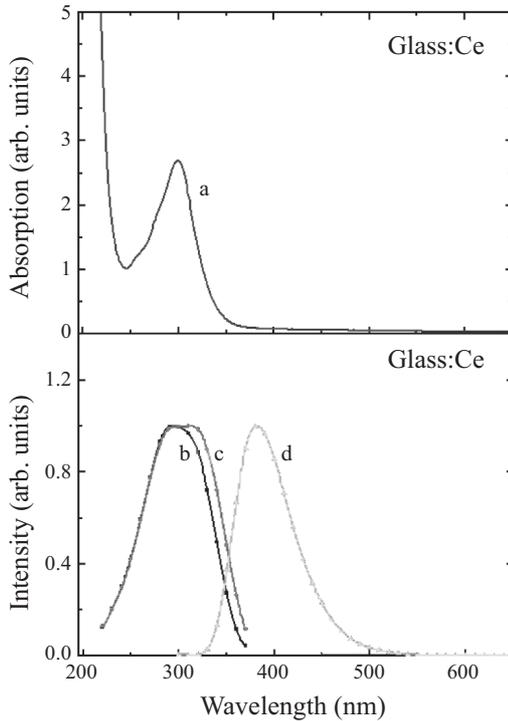


Fig. 1 Absorption (a), excitation ($\lambda_{em} = 380$ nm for curve b and $\lambda_{em} = 420$ nm for curve c) and emission ($\lambda_{ex} = 295$ nm, curve d) spectra of Ce^{3+} -doped silica glass sintered in a reducing (CO) atmosphere as measured at RT.

Characteristic broad emission band of Ce^{3+} is peaking around 375 nm under 295 nm excitation (curve d), but it is worth to note that the band is inhomogeneously broadened as its excitation spectrum shows low energy shift when emission wavelength is set longer (curve c). This is rather typical situation of Ce^{3+} center in glassy environment, in which the change in the short distance order and eventual defects nearby will shift position of $5d_1$ level and/or change its relaxation pathway. Usually,

the emission of Ce^{3+} ions occurs as doublet bands due to transitions from the relaxed lowest 5d excited state to the spin-orbit split 4f ground states ($^2F_{5/2}$, $^2F_{7/2}$). At RT, these two bands merge into an asymmetric broad band because of the large spatial extension of the 5d wave function and its interaction with lattice vibrations.

The PL decay curves of the Ce^{3+} -doped silica glass measured at RT are shown in Fig. 2. Each decay curve can be well fitted with a double-exponential equation, $I(t) = \sum A_i \exp(-t/\tau_i) + \text{background}$, $i = 1, 2$, where $I(t)$ is the luminescence intensity, A stands for the amplitude, t for the time and τ for the decay time constant of the exponential component. The PL decay at 370 nm under excitation at 290 nm (Fig.2a) yields the decay times $\tau_1 = 49.6$ ns and $\tau_2 = 147$ ns with respective component intensities $I_1 = (A_1\tau_1) / (\sum A_i\tau_i)$, namely, $I_1 = 93\%$ and $I_2 = 7\%$, respectively. The PL decay at longer wavelength of 440 nm under excitation with 320 nm (Fig.2b) yields the decay times $\tau_1 = 67.7$ ns and $\tau_2 = 255$ ns with respective component intensities $I_1 = 95\%$ and $I_2 = 5\%$, respectively. Increasing decay time value at longer emission wavelength is consistent with the above mentioned inhomogeneous broadening of Ce^{3+} emission band, indicating non-equivalent Ce^{3+} centers due to defects nearby, glassy host environment or similar reasons. While τ_1 decay times are typical for the $5d_1 \rightarrow 4f$ radiative transition of Ce^{3+} [14] there is no immediate explanation of longer τ_2 ones though respective component amplitude is very small.

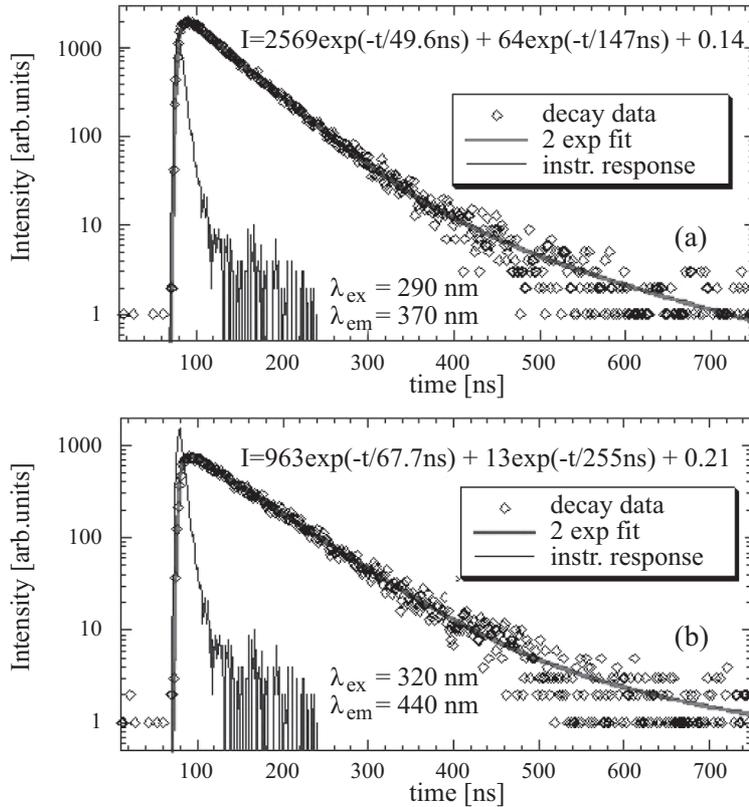


Fig. 2 PL decay curves of the Ce^{3+} -doped silica glass as measured at RT [(a) $\lambda_{\text{ex}} = 290 \text{ nm}$, $\lambda_{\text{em}} = 370 \text{ nm}$; (b) $\lambda_{\text{ex}} = 320 \text{ nm}$, $\lambda_{\text{em}} = 440 \text{ nm}$]. The solid lines are the convolution of the instrumental response and the function $I(t)$ given in the figures.

Somewhat non-exponential decay of Ce^{3+} center in Fig. 2 and varying τ_1 values with emission wavelength are worth a comment. Similar phenomena can be noticed in the silica glasses doped with Ce^{3+} ions [15, 16] and Eu^{2+} ions [3]: the $5d$ excited state of both Ce^{3+} and Eu^{2+} is sensitive to symmetry, short-range order and crystal field strength at a given site of host glass. Due to glass matrix, the dopant is embedded at a number of slightly non-equivalent sites, the emission band broadens inhomogeneously and the decay time value varies as well. Nevertheless, the decay non-exponentiality is rather small which means that the inhomogeneity of the Ce^{3+} sites is rather small.

3.2 Radioluminescence of Ce^{3+} -doped silica glass

RL spectra of a Ce^{3+} -doped silica glass and a BGO reference crystal recorded at RT are shown in Fig.3. The Ce glass shows a broad emission band peaking around 375 nm due to the $5d_1 \rightarrow 4f$ transitions of Ce^{3+} . Note a nearly coinciding position and shape for PL and RL spectra (Figs.1 and 3) of Ce glass. This result further confirms rather small inhomogeneity of the Ce^{3+} sites in the host glass. The Ce glass and BGO sample are about the same size and the measurements were performed in the same experimental conditions, so that the spectra can be compared in an absolute way. The integral scintillation efficiency (integral of RL spectra) of

the Ce glass is about 105% of that of the BGO scintillator, determined from the ratio of its integrated RL intensity to that of the BGO reference sample.

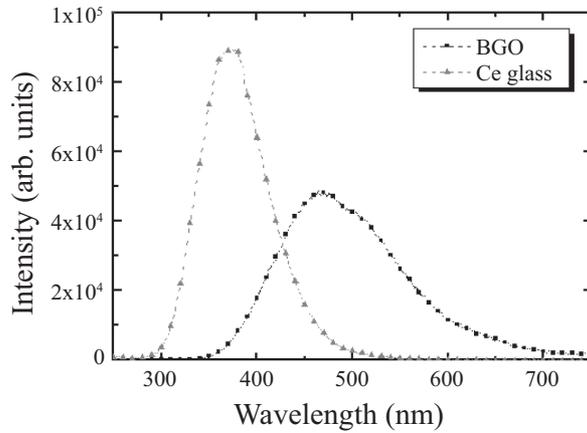


Fig. 3 RL spectra (X-ray excitation: 40 kV, 15 mA) of Ce^{3+} -doped silica glass and BGO crystal as measured at RT. The spectra are mutually comparable in an absolute way.

LY measurements were performed by measuring pulse height spectra of 662 keV γ -rays from a ^{137}Cs source. The sample was coupled by silicone grease to the Photonis XP5200B PMT and covered with several layers of Teflon tape in a

configuration of a reflective umbrella for better light collection. Fig.4 presents the pulse height spectra of 662 keV γ -rays from a ^{137}Cs source as measured with Ce glass, CeF_3 and BGO crystals, but with lower amplification gain factor of 0.18 for BGO.

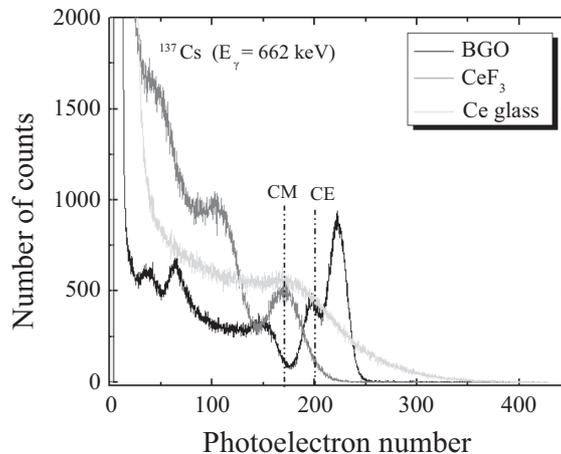


Fig. 4 Pulse height spectra of 662 keV γ -rays from a ^{137}Cs source as measured with Ce glass, CeF_3 and BGO (with a lower gain factor of 0.18) crystals.

The intensity in a scintillation pulse (x-axis), expressed as a number of photoelectrons released from the PMT photocathode, was obtained by relating the position of a photopeak of 662 keV γ -rays detected in the CeF_3 crystal with that of the single photoelectron peak from the PMT photocathode [17,18]. We obtained photoelectron yield of 1850 and 260 photoelectrons per MeV (phe/MeV) for BGO and CeF_3 crystals, respectively. By taking into account the quantum efficiency of the PMT for the BGO (21% at peak emission 480 nm) and CeF_3 (6% at peak emission 290 nm), we estimated the LY of 8810, and 4330 photons/MeV (ph/MeV), respectively, for BGO and CeF_3 crystals which agrees well with the earlier reported values [18,19]. Unfortunately, Ce glass shows only the Compton continuum (CC) with the absence of 662 keV photopeak in the pulse height spectrum. This is due to low density and effective atomic number of the glass matrix. However, we can estimate the LY of Ce glass using the position of Compton edge (CE), which corresponds to a maximum energy of Compton electron ($E_e = 477$ keV), in the pulse height spectrum of 662 keV γ -rays. It must be noted that exact position of CE in the CC is not located at the

same position with Compton maximum (CM) but it slightly shifts to higher energy shoulder [20]. A rough estimate of the CE position for a low density glass scintillator can be obtained by assuming a value of 0.8 [20] for a fraction of CM at which CE crosses the CC spectrum (see Fig.4). We estimated the photoelectron yield of 420 phe/MeV (at $E_e = 477$ keV). This value corresponds to the LY of 1400 ph/MeV by taking into account a quantum efficiency of 30% for the PMT at peak emission (375 nm) of the Ce glass. In spite of comparable integral scintillation efficiency under X-ray irradiation ($E_x \leq 40$ keV), the scintillation LY of the Ce glass under excitation with high energy (662 keV) gamma rays is much lower i.e. about 16% of that of BGO crystal (as measured at integrating time of 4 μ s). This indicates to a strong slow-down effect in the transport stage of scintillation mechanism in the Ce glass as its photoluminescence decay time is much shorter (50-70 ns, Fig. 2) than that of BGO (300 ns). Taken together, this effect and short-range order of glass matrix demonstrate that energy transfer efficiency of scintillating glasses is much lower with respect to single crystal materials, thus reducing scintillation LY of Ce glass.

Table 1 Photoelectron yield and light yield for Ce^{3+} -doped silica glass, CeF_3 and BGO crystals as measured using ^{137}Cs source.

Scintillator	Photoelectron yield [phe/MeV]	Light yield [ph/MeV]
BGO	1850 \pm 90	8810 \pm 880
CeF_3	260 \pm 10	4330 \pm 430
Ce glass	420 \pm 20	1400 \pm 140

4. Conclusions

The Ce³⁺-doped silica glass with small inhomogeneity of Ce sites was successfully prepared by impregnation of Ce(NO₃)₃ solution into a porous silica glass and sintered at high temperature in a CO reducing atmosphere. It exhibits an intense violet-blue emission of Ce³⁺ ions peaking around 375 nm under UV and X-ray excitation with a fast luminescence decay time of about 50 ns. Moreover, it shows a high integral scintillation efficiency under X-ray irradiation comparable to that of the BGO crystal while the LY of about 16% of that of the BGO is obtained under excitation with 662 keV γ -rays. In terms of the overall scintillation efficiency, this Ce³⁺-doped silica glass prepared by such a unique technology appears to be clearly superior than the Ce³⁺-doped glass prepared by direct high-temperature melting of rare-earth compounds and raw materials of glass[21]. It seems worthwhile to enhance the scintillation efficiency further e.g. by optimizing the concentrations of Ce³⁺ and Gd³⁺ co-doping in the silica glass, which employs the efficient energy transfer from Gd³⁺ to Ce³⁺. It could be useful for developing new UV and X-ray sensors or phosphors.

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