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Spectroscopic Properties of Nd^{3+} and Pr^{3+} in Gallate Glasses with Low-Phonon Energies

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Abstract

Absorption and fluorescence spectra of Nd^{3+} were measured in potassium tantalum gallate (KTG), lead bismuth gallate (PBG), ZBLAN and Ge-Ga-S glasses. Judd-Ofelt analysis was performed to determine the spontaneous emission probability and stimulated emission cross section of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+}. Raman spectra were studied to clarify maximum phonon energies of the glasses. We observed the fluorescence of the $^1G_4 \rightarrow {}^3H_5$ transition of Pr^{3+} in a dehydrated PBG glass for the first time. The PBG glass has a higher quantum efficiency than that of ZBLAN glass based on Judd-Ofelt analysis.

Key words: laser materials, gallate glass, Judd-Ofelt analysis

Glasses with low-phonon energies are of particular interest for the hosts of infrared and infrared-to-visible upconversion lasers and 1.3 μm optical fibre amplifiers[1]. The maximum phonon energies of conventional oxide glasses such as silicate, borate and phosphate glasses are high therefore the non-radiative relaxation due to the multiphonon relaxation is the dominant process for the transitions with small energy gaps, e. g. ~ 3000 cm^{-1} for $^1G_4$ - $^3H_5$ of Pr^{3+}. Since halide[2] and chalcogenide[1,3] glasses have considerably lower phonon energies than the oxide glasses, these non-oxide glasses have been studied mainly as the hosts for the above applications. On the other hand, there are some oxide glasses with low phonon energies such as tellurite and gallate glasses; in which upconversion fluorescence of Er^{3+} was observed even in these glasses[4].

Judd-Ofelt analysis has been used to calculate the spontaneous emission probabilities and stimulated emission cross sections for rare-earth ions to clarify the compositional dependence of the optical properties. According to Judd-Ofelt theory[5,6], three intensity parameters Ω_t (t=2,4,6) associated with glass composition are determined. It is known for glasses that the JO parameters are related to local structures around rare-earth ions and/or the covalency of the rare-earth ion sites[7]. Trivalent neodymium ions have been investigated in many types of glasses to discuss the optical properties of rare-earth ion doped glasses for laser hosts. A previous study[7] revealed that Judd-Ofelt analysis for Nd^{3+} is rather reliable, compared with those of other rare-earth ions; even if the type of rare-earth ion varies from Pr^{3+} to Tm^{3+}, the compositional dependence of the JO parameters are the same for all the glass systems. There are several studies on the compositional dependence of the JO parameters in tellurite glasses[8]; to our knowledge, there are few reports on the JO parameters of Nd^{3+} in gallate glasses. Although previous studies reported the JO parameters of Er^{3+} in an alkali alkaline-earth gallate glass, they pointed out that the gallate glass is not a practical laser host due to its poor chemical durability[9].

In this work absorption and fluorescence of Nd^{3+} in tantalum gallate and heavy metal gallate glasses with relatively good chemical durability were measured and analyzed using

Judd-Ofelt theory to reveal the features of the gallate glasses and compare these properties with typical non-oxide glasses, i. e., ZBLAN[2] and $Ge_{25}Ga_5S_{70}$[3]. Oxide glasses generally have advantages of thermal stability and easy mass production at low cost, as compared with the non-oxide glasses; however, oxide glasses possess relatively higher phonon energies. The latter feature degrades emission properties due to the multi-phonon relaxation in the transitions with small gaps. The another purpose of this study is to demonstrate that the heavy metal gallate glass is a possible candidate for a low-cost praseodymium-doped fibre amplifier (PDFA) and an alternative to the ZBLAN glass, whose fibers are currently used for this application.

Two types of gallate glasses with compositions of $40K_2O{\cdot}20Ta_2O_5{\cdot}40Ga_2O_3$ (KTG) and $60PbO{\cdot}20Bi_2O_3{\cdot}20Ga_2O_3$ (PBG) in molar ratio were prepared using conventional melting techniques. These glasses have good chemical durability in air and their surface qualities have been unchanged under an ambient atmosphere even after three months. Glass transition and crystallization temperatures are 695° and 806°C for KTG and 348° and 451°C for PBG; the difference between these temperatures is over 100°C, which suggests the possibility of drawing fibres successfully. A previous study reported the successful fibre drawing of PBG glass[10]. A fluoride glass with a composition of $53ZrF_4{\cdot}20BaF_2{\cdot}4LaF_3{\cdot}3AlF_3{\cdot}20NaF$ (ZBLAN) was prepared in Ar using a gold crucible. A sulfide glass with a composition of $Ge_{25}Ga_5S_{70}$ (GGS) was melted in an evacuated silica ampoule using refined raw materials. The glasses were doped with 0.3 mol% Nd_2O_3 for KTG, 0.2 mol% Nd_2O_3 for PBG, 0.6 mol% NdF_3 for ZBLAN, and 0.12 at% Nd^{3+} for GGS. Absorption spectra were recorded with a Hitachi U-3500 spectrophotometer in the range of 400 to 1000 nm. Refractive indexes as a function of wavelength were measured with an Atago 1T Abbe refractometer for ZBLAN glass and a Kalnew GMR-1 precision spectrometer for KTG, PBG and GGS glasses. Density was determined by Archimedes method using kerosene as an immersion liquid. The concentration of Nd^{3+} was calculated from the densities and batch compositions of samples. Fluorescence spectra of the $^4F_{3/2} \rightarrow$

$^4I_{11/2}$ transition were measured with a Spectra Diode Labs. SDL-5412-H1 laser diode as a pump source at 800 nm and with a Ge detector in the range of 1000 to 1200 nm. A Xe flash lamp and a Ge detector were used for fluorescence lifetime measurements. The JO parameters, spontaneous emission probability and stimulated emission cross section for Nd^{3+} were calculated using the method adopted by Krupke[11]. Raman spectra were measured with an argon-ion laser pump at 514.5 nm and with a triple-grating spectrometer and liquid nitrogen-cooled CCD array.

Figure 1 shows absorption spectra of Nd^{3+} in KTG, PBG, ZBLAN and GGS glasses. The peak wavelengths of all the transitions shift to longer wavelengths in the order ZBLAN < KTG ~ PBG < GGS. This shift is due to the nephelauxetic effect caused by the difference of the ligand for Nd^{3+}, i. e., S^{2-}, O^{2-}, F^-. The absorption cross section of the hypersensitive $^4I_{9/2} \rightarrow {}^4G_{5/2}, {}^2G_{7/2}$ transition numbered 5 is much higher than the other transitions in KTG and GGS glasses. This feature corresponds to the magnitude of the JO parameter Ω_2. As shown in Table 1, KTG and GGS glasses have considerably higher Ω_2 values compared with PBG and ZBLAN glasses. The Ω_2 parameter is most sensitive to glass composition; it is affected by the covalency of the ligand and local structures around Nd^{3+} ions[3,7]. The reason for GGS glass may be based on the covalent character in bonding between Nd and S, corresponding to the nephelauxetic effect. However, that for KTG is believed to be the asymmetry around Nd^{3+} ions, which is related to the mixed anions structure of Ga_2O_3 and Ta_2O_5[12]. Figure 2 shows fluorescence spectra of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition in the glasses. The peak wavelength of the fluorescence shifts to longer wavelengths in the same order of those of the absorption spectra shown in Fig. 1. The fluorescence of KTG glass has the broadest band in all the glasses because of its mixed anions structure.

Table 1 summarizes the results of Judd-Ofelt analysis of Nd^{3+} for KTG, PBG, ZBLAN and GGS glasses. The largest Ω_2 of 11.4 pm^2 is obtained in the tantalum gallate glass. Ω_4 is on the order GGS > KTG ~ PBG > ZBLAN; Ω_6 is on the order GGS > KTG >

ZBLAN > PBG. The spontaneous emission probability and stimulated emission cross section are calculated based on the Judd-Ofelt theory[11]

$$A[(S, L)J:(S', L')J'] = \frac{64\pi^4 e^2 n}{3h(2J+1)\bar{\lambda}^3}\left[\frac{(n^2+2)^2}{9}\right] \times \sum_{t=2,4,6} \Omega_t \left|\langle\langle (S, L)J \| U^{(t)} \| (S', L')J' \rangle\rangle\right|^2 \quad (1),$$

$$\sigma_p = \frac{\lambda_p^4}{8\pi c n^2} \frac{A[^4F_{3/2} \rightarrow {}^4I_{11/2}]}{\Delta\lambda_{eff}} \quad (2),$$

where λ is the mean wavelength of the fluorescence band, J is the total angular momentum of the initial level, n is the refractive index at the mean wavelength, $< \|U^{(t)}\| >$ are the reduced matrix elements calculated in the intermediate-coupling approximation, $\Delta\lambda_{eff}$ is the effective fluorescence linewidth. The spontaneous emission probability A of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+} (~ 1.06 μm) is determined three terms: n and Ω_4 and Ω_6 parameters because of $U^{(2)} = 0$, $U^{(4)} = 0.1423$, $U^{(6)} = 0.4070$ for the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+} [11]; the stimulated cross section σ_p at its peak wavelength λ_p is related to A and $\Delta\lambda_{eff}$. GGS glass exhibits the highest A and σ_p in all the glasses examined because of the highest refractive index and highest Ω_4 and Ω_6 and narrowest $\Delta\lambda_{eff}$. KTG also has a high A value because of high Ω_4 and Ω_6; however σ_p is not very large because of the broader $\Delta\lambda_{eff}$. PBG glass has higher A and σ_p than those of KTG and ZBLAN glasses because of higher n and Ω_4 and Ω_6; these values of the PBG glass are comparable to those of tellurite glasses[8]. The measured fluorescence lifetime τ_m is varied in the range of 96 μs (for GGS) to 494 μs (for ZBLAN); this order is related to the reciprocal spontaneous emission probability. If the multiphonon relaxation is negligibly small in the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition, the quantum efficiency defined as $\eta \equiv \tau_m/\tau_c$, where τ_c is the calculated radiative lifetime, should be close to 100%. η is larger than 100% within 25% for KTG, PBG and GGS glasses. Such a discrepancy was also observed in tellurite glasses[8]; its reason may be caused by the result that the calculated line strength of the $^4I_{9/2} \rightarrow {}^4F_{3/2}$ transition is larger than the measured line strength[11], as they pointed out.

In order to discuss the possibility of applying the gallate glasses to 1.3 μm optical fibre amplifiers, Raman spectra of the glasses were measured. Figure 3 shows Raman spectra of KTG, PBG, ZBLAN and GGS glasses. GGS glass has the lowest phonon energy with a main peak at 350 cm^{-1} in all the glasses. PBG glass has a comparable low phonon energy with ZBLAN glass with a maximum phonon energy of 550 cm^{-1}. This energy is related to the vibrational mode of Ga - O tetrahedra with four-coordinated Ga^{3+} ions[13]. PBG glass also shows a broad absorption band spreading up to ~ 750 cm^{-1}. This broad absorption band may correspond to the vibration of Ga - O with bridging oxygens (at 550 cm^{-1}) and non-bridging oxygens (at 650 cm^{-1})[13]. KTG glass has relatively higher phonon energy with a maximum of 900 cm^{-1} due to Ta - O - Ga vibration. Since the quantum efficiency of the $^1G_4$ - $^3H_5$ of Pr^{3+} is dominated by the multiphonon relaxation due to the vibration of glass networks[1], PBG glass is expected to be possible for a host of 1.3 μm optical fibre amplifiers. It offers a high quantum efficiency because of higher spontaneous emission probability and a phonon energy similar to that of the ZBLAN glass. If the peak wavelength of the $^1G_4$ - $^3H_5$ of Pr^{3+} shifts with the same order of that of $^4I_{9/2} \rightarrow {}^4F_{3/2}$ of Nd^{3+}, as shown in Fig. 2, the PBG glass may be also a preferable host with a high gain in the 1.3 μm telecommunications window[1].

Except for tellurite glasses[14], the fluorescence of the $^1G_4 \rightarrow {}^3H_5$ transition of Pr^{3+} has not been observed in any oxide glasses. PBG glass melted in air contains a large amount of OH with a high phonon energy of ~ 3000 cm^{-1}; the retaining OH affects significantly the $^1G_4 \rightarrow {}^3H_5$ transition of Pr^{3+}; no fluorescence was observed in the PBG glass melted in air. We successfully prepared a PBG glass with a low OH concentration (a few ppm in weight) by the substitution of PbF_2 for a part of PbO and melting in dried O_2 with a water vapor pressure of 10 Pa.

Figure 4 shows the fluorescence spectrum of Pr^{3+} in the PBG glass with a low OH concentration. The concentration of Pr^{3+} ions was 500 ppm in weight. The measured fluorescence lifetime which was fitted to a stretched exponential was 292 μs. The fluorescence was measured by a excitation at 1010 nm using a Ti: sapphire laser. The quantum efficiency η calculated by Quimby method based on relative fluorescence measurements[15] is 7%; that by Judd-Ofelt theory defined as τ_m/τ_R , where τ_m is the measured fluorescence lifetime and τ_R the radiative lifetime (the reciprocal total radiative transition probability, A^{-1}) is 36%. Table 2 summarizes the JO parameters of Pr^{3+}, radiative lifetime τ_R from the $^1G_4$ level, measured fluorescence lifetime τ_m of the $^1G_4 \rightarrow {}^3H_5$ transition for the PBG, ZBLAN[2], Ga-La-S[16] and Ge-Ga-S[3, 17] glasses. The values of η for the PBG glass are higher than 4% for ZBLAN glass[2]. Further experiments are needed for the confirmation of the higher quantum efficiency of the PBG glass. Although sulfide glasses have much higher quantum efficiencies, the PBG glass has an advantage in its easy fiber fabrication process and may be a candidate for a low-cost PDFA for optical telecommunications.

In summary, we have studied absorption and fluorescence properties of Nd^{3+} and analyzed based on Judd-Ofelt theory for potassium tantalum and heavy metal gallate glasses in comparison with ZBLAN and Ge-Ga-S glasses. The potassium tantalum gallate glass has the highest Ω_2 value in the glasses and relatively high Ω_4 and Ω_6 and high spontaneous emission probability A of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition. The lead bismuth gallate glass exhibits high A and stimulated emission cross section because of its high refractive index; it has a comparable low phonon energy with ZBLAN glass. The PBG glass possesses a higher radiative transition probability from the $^1G_4$ level of Pr^{3+} in comparison with the ZBLAN glass. We observed the fluorescence of the $^1G_4 \rightarrow {}^3H_5$ transition of Pr^{3+} in the dehydrated PBG glass for the first time. The PBG glass has a higher quantum efficiency than that of ZBLAN glass. The PBG glass is a candidate for the Pr^{3+} doped low-cost optical fiber amplifier in the 1.3 μm telecommunication window.

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Figure captions

Fig.1 Absorption spectra of Nd^{3+} in (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses. All transitions are from the $^4I_{9/2}$ level to the following levels: (1) $^4F_{3/2}$, (2) $^4F_{5/2}$, $^2H_{9/2}$, (3) $^4F_{7/2}$, $^4S_{3/2}$, (4) $^4F_{9/2}$, (5) $^4G_{5/2}$, $^2G_{7/2}$, (6) $^2K_{13/2}$, $^4G_{7/2}$, $^4G_{9/2}$, (7) $^4G_{11/2}$, $^2G_{9/2}$, $^2D_{3/2}$, $^2K_{15/2}$ and (8) $^2P_{1/2}$, $^2D_{5/2}$.

Fig. 2 $^4F_{3/2} \rightarrow {}^4I_{11/2}$ fluorescence spectra of Nd^{3+} in (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses.

Fig. 3 Raman spectra of (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses.

Fig. 4 Fluorescence spectrum of Pr^{3+} in the dehydrated PBG glass.

Table 1 Radiative properties of Nd^{3+} in KTG, PBG, ZBLAN, and GGS glasses.

Glass host composition (mol%)	Ω_2	Ω_4 (pm²)	Ω_6	n_d	A* (s^{-1})					τ_c (μs)	τ_m (μs)	η (%)	λ_p (nm)	$\Delta\lambda_{eff}$ (nm)	σ_s (pm²)
					$^4I_{9/2}$	$^4I_{11/2}$	$^4I_{13/2}$	$^4I_{15/2}$	A_{tot}						
$40K_2O \cdot 20Ta_2O_5 \cdot 40Ga_2O_3$	11.4	5.04	5.57	1.741	1841	2115	401	21	4376	229	223	97.7	1071	41.1	3.06
$60PbO \cdot 20Bi_2O_3 \cdot 20Ga_2O_3$	3.72	4.48	4.33	2.346	4293	4449	795	38	9574	104	129	124	1071	33.0	4.71
ZBLAN	2.37	3.77	4.85	1.475	894	1126	223	11	2254	444	494	111	1056	26.3	3.17
$Ge_{25}Ga_5S_{70}$	10.6	9.56	6.04	2.175	6543	5471	865	41	12920	77.4	96.0	124	1084	26.2	8.73

*Spontaneous emission probabilities from the $^4F_{3/2}$ level.

Table 2 Judd-Ofelt parameters of Pr^{3+}, radiative lifetime from the $^1G_4$ level, measured fluorescence lifetime and quantum efficiency of $^1G_4 \rightarrow {}^3H_5$ for low-phonon energy glasses.

Glass host	Ω_2	Ω_4	Ω_6	τ_R	τ_m	η
	(pm^2)			(μs)	(μs)	(%)
PBG	4.0	6.0	3.7	805	292	36
ZBLAN [2]	1.43	4.22	4.87	3248	110	3.4
Ga-La-S [16]	7.3	6.2	3.9	510	295	58
Ge-Ga-S [3]	12.8	4.3	7.7	511	360	70
Ge-Ga-S [17]	12	7.3	6.0	604	299	50

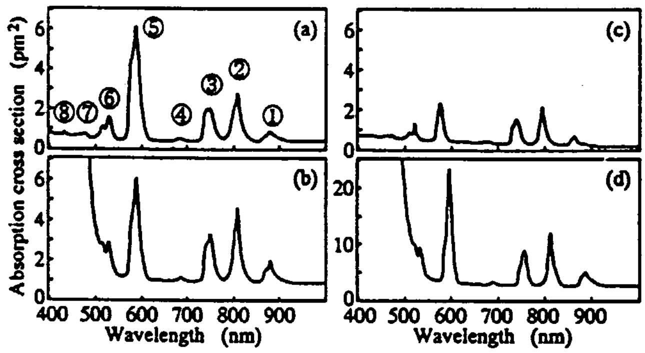

Fig. 1 Absorption spectra of Nd^{3+} in (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses. All transitions are from the $^4I_{9/2}$ level to the following levels: ① $^4F_{3/2}$, ② $^4F_{5/2}$, $^2H_{9/2}$, ③ $^4F_{7/2}$, $^4S_{3/2}$, ④ $^4F_{9/2}$, ⑤ $^4G_{5/2}$, $^2G_{7/2}$, ⑥ $^2K_{13/2}$, $^4G_{7/2}$, $^4G_{9/2}$, ⑦ $^4G_{11/2}$, $^2G_{9/2}$, $^2D_{3/2}$, $^2K_{15/2}$, and ⑧ $^2P_{1/2}$, $^2D_{5/2}$.

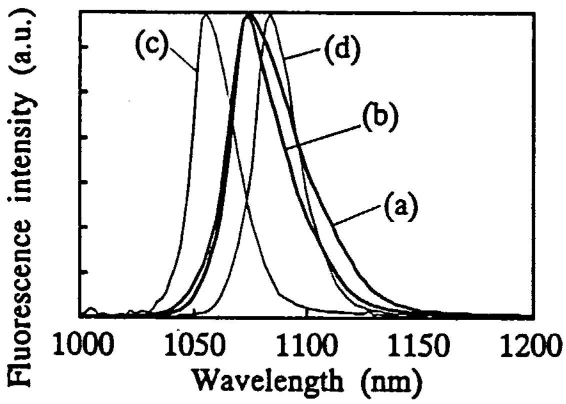

Fig. 2 $^4F_{3/2} \rightarrow {}^4I_{11/2}$ fluorescence spectra of Nd^{3+} in (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses.

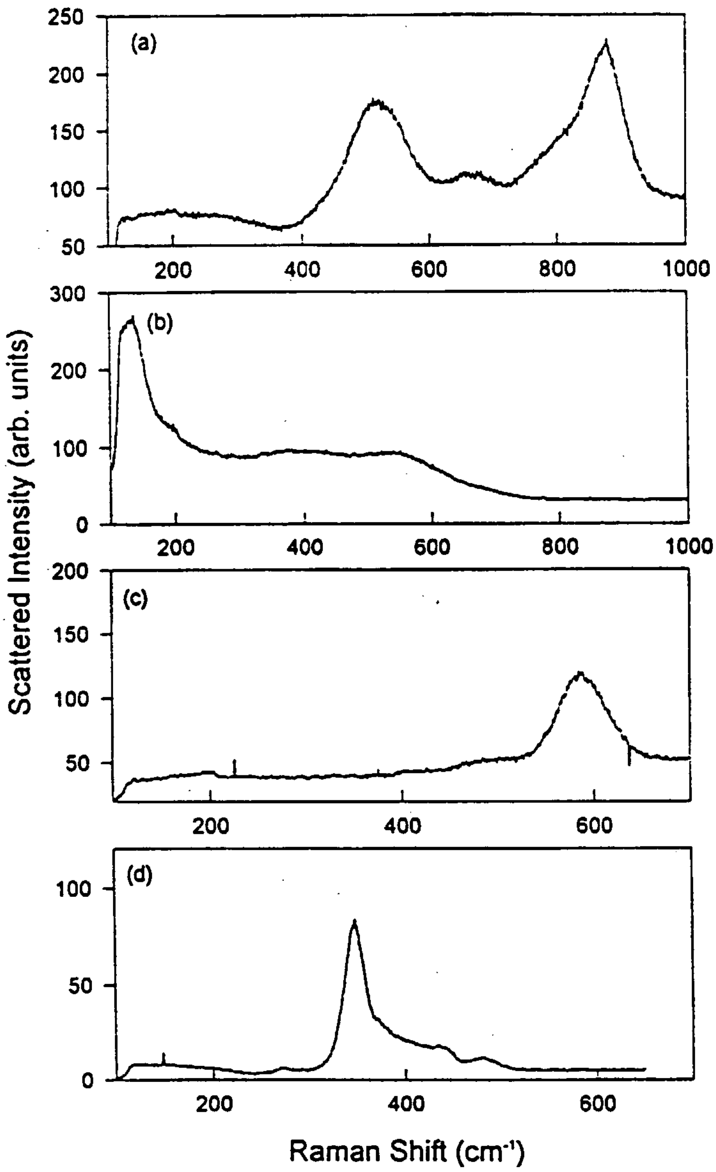

Fig.3 Raman spectra of (a) KTG, (b) PBG, (c) ZBLAN, and (d) GGS glasses.

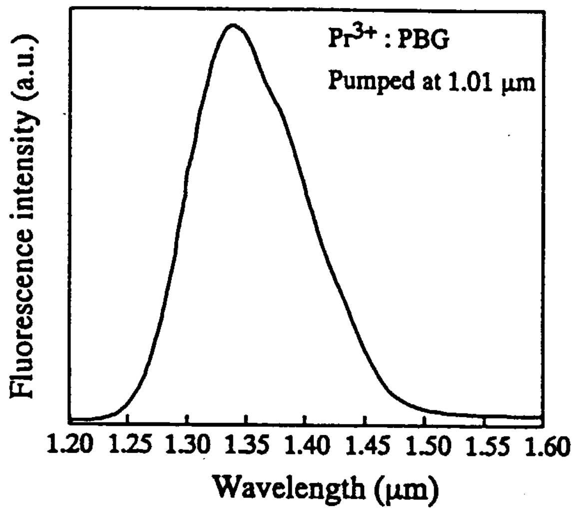

Fig. 4 Fluorescenice spectrum of Pr^{3+} in the dehydrated PBG glass.