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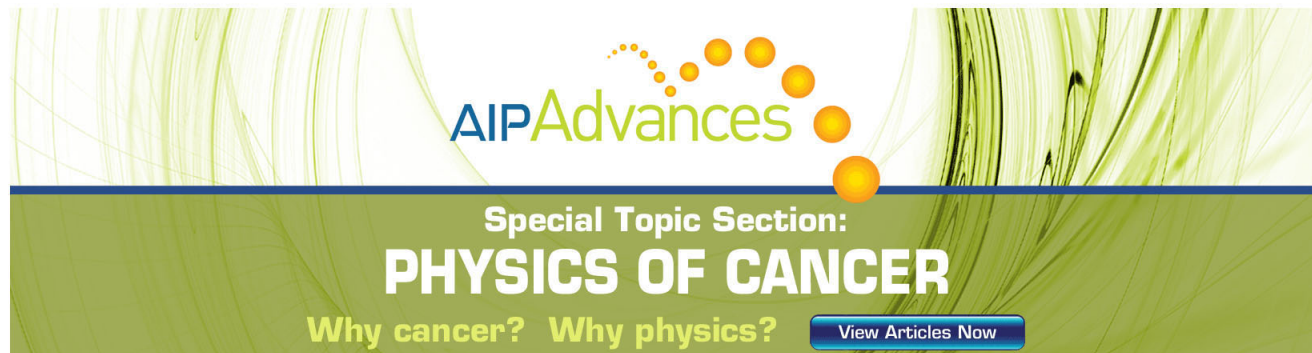
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Fluorescence properties of divalent and trivalent europium ions in aluminosilicate glasses

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Optical absorption, fluorescence, excitation and time-resolved spectra, and fluorescence dynamics were studied in Eu^{2+}- and Eu^{3+}-codoped aluminosilicate glasses. The results indicated that the energy transfer occurred between Eu^{2+} and Eu^{3+} ions, in which the energy corresponding to the $4f^6 5d$ to $^8S_{7/2}$ transition of Eu^{2+} ions caused excitation of the Eu^{3+} ions to the ^5D_J level. Some of the electrons in the ground state $^7F_0$ were thermally excited into the $^7F_1$ state at room temperature, and thermal populations of the $^7F_1$ level had great effects on the spectral shape of the $^5D_0-^7F_2$ emission. The effect of light irradiation at different wavelengths on fluorescence was systemically studied. The visible, ultraviolet and x-ray irradiation caused a decrease in the fluorescence of Eu^{2+} and/or Eu^{3+} ions. Frequency selective excitation experiments were performed at 10 K and room temperature. Instead of forming a spectral hole, the intensity of the $^7F_0-^5D_0$ transition decreased as a whole. This was attributed to the interaction of Eu^{3+} ions with the two-level systems existing in the glass network. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1450052]

I. INTRODUCTION

Rare earth ions energetically prefer the trivalent state, of which the $4f^n$ configuration is relatively isolated and the next excited $4f^{n-1}5d$ configuration is situated in the high energy level above the ground state of the $4f^n$ configuration. According to this electron configuration, the sharp lines due to the $f-f$ transitions are observed in the optical absorption and emission spectra. Among them, Eu ions as well as Er, Yb, and Sm ions have attracted significant attention, because they have a large potential for applications in some optical fields, such as lasers, fiber amplifiers, phosphors, electroluminescent devices, and high-density optical storage.[1–7] The Eu^{3+} ions, of which the lowest excited level ($^5D_0$) of the $4f^6$ configuration is situated below the $4f^5 5d$ configuration, show very sharp emission lines extending from visible to the near-infrared. The persistent spectral hole burning (PSHB) can be performed in the $^7F_0-^5D_0$ transition of Eu^{3+} ions.[8–10] It is well known that PSHB has potential application as a frequency-domain storage media and is an effective technique for studying the local environment and the low temperature dynamics of glasses and other amorphous solids.[11–13] Recently, room temperature PSHB was observed in Eu^{3+}-doped oxide glasses.[9,10,14,15] The PSHB mechanism of Eu^{3+} ions in the oxide glasses is not clear. Generally, it is believed that the Eu^{3+} ions at the burned sites are changed into Eu^{2+} ions through PSHB, although conclusive evidence may be lacking.[10] In the Eu^{3+}-doped fluoride mixed crystal, it is clear that the spectral hole is formed by the site change of Eu^{3+} ions among different local environments.[16]

The Eu^{2+} ions have complex energy levels, which in turn is modified by the host matrices. The first excited $4f^6 5d$ configuration lies close to the excited $4f^7$ levels. The transitions between the first excited $4f^6 5d$ configuration and the $4f^7$ ground state are dipole allowed and give a very intense emission intensity about 10^6 times those of the $f-f$ transitions in the trivalent ions. Furthermore, because of the large spatial extension of the $5d$ wave function, the optical spectra due to the $f-d$ transitions are usually broadened and depend on the surroundings of the Eu^{2+} ions. Thus, the choice of host is a critical parameter for determining the optical properties of the Eu^{2+} ions.[17] Eu^{2+} ions in the host of the BaFCl family is a favorable photostimulated luminescent material. Very recently, photostimulated luminescence was observed in Eu^{2+} ion doped fluoride glasses.[18] Long-lasting phosphorescence was also reported in Eu^{2+} and Eu^{3+}-codoped aluminosilicate glasses by Qiu *et al.*[19] In fact, PSHB of Eu^{2+} ions also attracts much interests due to its high hole-burning density.[20,21]

Oxide glasses have proven useful as host materials for rare earth elements because of their high transparency, compositional variety, and easy mass production. Recently, we successfully prepared Eu-doped aluminosilicate glasses, which had strong emissions of Eu^{2+} ions and Eu^{3+} ions. It should be noted that the glasses prepared were not hole burned even at a temperature as low as 10 K. Actually, very strong optical bleaching was observed in the glasses. In this article, we demonstrate the fluorescence behavior in Eu^{2+} and Eu^{3+}-codped aluminosilicate glasses. The analysis of the fluorescence behavior is needed to clarify the hole burning mechanism in the host of glasses and to provide a guide for a new host with strong emission and good PSHB behavior.

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TABLE I. Component of the aluminosilicate glasses in wt. %.

SiO_2	Al_2O_3	Li_2O	MgO	Na_2O	TiO_2	ZrO_2	P_2O_5	Sb_2O_3
60–70	20–30	5–10	2–4	1–2	2–5	2–7	2–10	0–2

II. EXPERIMENTS

A. Glass preparation

The aluminosilicate glasses normally containing 0, 0.5, 1, and 2 wt % Eu_2O_3 were prepared by the method of solid state reaction. The glass compositions are listed in Table I. All of the starting materials (with purity of 99.9%) were commercially available. In the preparation, they were mixed together and stirred until the mixture became homogeneous. The mixture was then melted in the air. After keeping at 1450 °C for 4 h and slowly cooling the melt to room temperature, colorless transparent glasses were formed. The glass samples were cut into small pieces (1 cm×1 cm) with thickness of 1 mm and were well polished. After x-ray irradiation, the color of the glasses became dark red.

B. Spectral measurements

The absorption, excitation, and fluorescence spectra were all measured with a Hitachi F-4500 fluorescence spectrometer with a resolution of 1 nm. The absorption spectra were measured by the sample-in, sample-out method. The measurements of the time-resolved spectra and the fluorescence dynamics used a 266 nm pulsed Nd:YAG (aluminum–garnet) laser (with a linewidth of 0.2 cm^{-1}, pulse duration of 10 ns, and repetition frequency of 10 Hz) combined with a fourth harmonic generator as the pump. The resonance excitation used a Rhodamine 6G dye laser pumped by the same Nd-YAG laser. The same laser light fixed at 578 nm was used as PSHB, which was also called frequency-selective excitation. A boxcar and a Spex 1403 spectrometer were used for detection. In the measurements, the glasses were put into a helium-gas-cycling system, in which the temperature varied from 10 to 300 K.
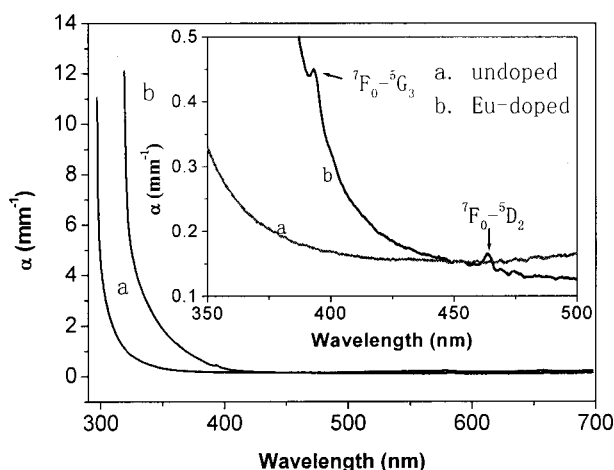
III. RESULTS AND DISCUSSION

A. Absorption, fluorescence, and excitation spectra

The optical absorption spectra of the Eu-doped glass in comparison with the undoped aluminosilicate glass are shown as Fig. 1. In the visible range (400–700 nm), the absorption coefficient α, was nearly constant with wavelengths for both glasses. In the ultraviolet range, α increased significantly as the wavelength decreased. Compared to the host glass, the absorption limit of the Eu-doped glass shifted to redside, indicating that the absorption caused by Eu doping occurred shorter than 400 nm. The inset in Fig. 1 shows two sharp absorption peaks at 393 and 464 nm in the Eu-doped glass, which were attributed to the $^7F_0-^5G_3$ and the $^7F_0-^5D_2$ transition of Eu^{3+} ions, respectively. The $f-f$ absorption cross section of Eu^{3+} ions in oxide glasses is generally very weak. After x-ray irradiation, the absorption limit shifted to the red side, while the absorption amplitude caused by the $f-f$ transition of Eu^{3+} ions did not change.

Figure 2 shows the fluorescence spectrum of the Eu^{2+} and Eu^{3+} ions in the aluminosilicate glass. A broad emission band associated with the transition of $4f^7-4f^65d$ of the Eu^{2+} ion was observed in the glass. The sharp lines of $^5D_0-^7F_J$ of Eu^{3+} ions were also observed. Among them, the emission of $^5D_0-^7F_2$ was strongest. The transitions of ^5D_J ($J=1,2,4$), $^5G_3$, and $^5L_6$ to the ground states ^7F_J were not clearly observed because they were weak and mashed by the $f-d$ transition of Eu^{2+} ions.

Figure 3 shows the excitation spectrum of the Eu-doped glass. A broadband having a peak at 240 nm was observed, which probably originated from the transition of the charge-
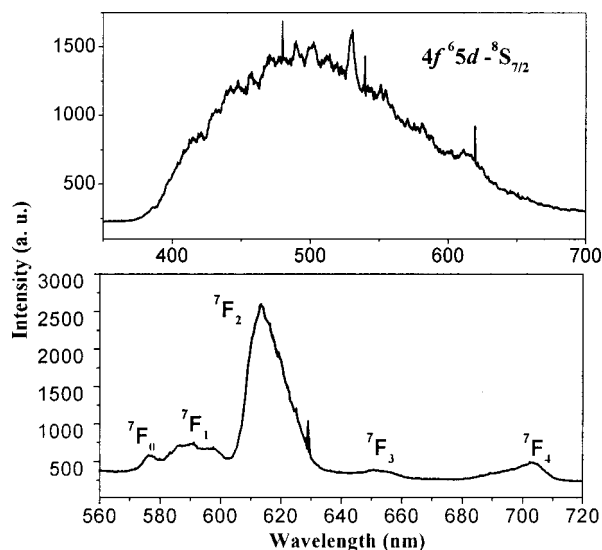

FIG. 1. Optical absorption spectra in one undoped and one Eu-doped (2 wt %) alumino-silicate glass. (Inset) The same spectra in a small wavelength scale.

FIG. 2. Fluorescence spectra of Eu^{2+} (a) and Eu^{3+} (b) in the Eu-doped aluminosilicate glass (2 wt %, This glass is used as a typical one). The spectra were measured under the pump of a 266 nm pulsed laser.
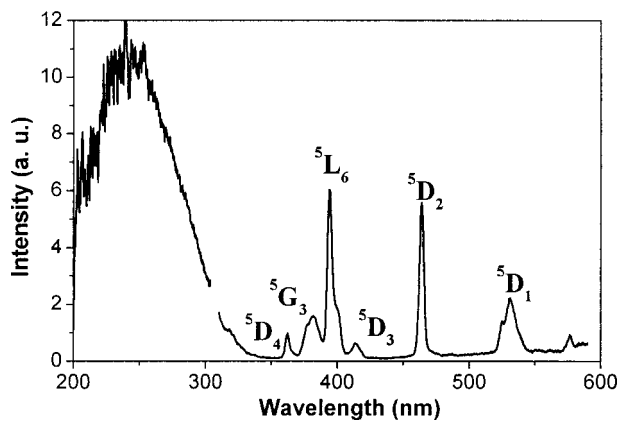
FIG. 3. Excitation spectrum of the Eu-doped aluminosilicate glass within the $^5D_0-^7F_2$ transition of Eu^{3+} ions. The monitor wavelength is at 614 nm.

transfer state of the Eu^{3+} ion. Sharp lines were observed at 340, 362, 387, 393, 414, 464, and 531 nm, respectively, These lines are associated with the $f-f$ transitions of Eu^{3+} ions. Their detailed origins are labeled in Fig. 3.

B. The evidence of energy transfer between Eu^{2+} and Eu^{3+}

Figure 4 shows the fluorescence dynamics of Eu^{2+} on the μs time scale. The experimental dynamics was well fitted by biexponential functions. The two exponential decay constants were determined to be 0.30 ± 0.03 and 2.8 ± 0.2 μs by fitting. Here we did not observe an increase in Eu^{2+} fluorescence due to the instrumental limitation. We estimate the increasing time constant to be shorter than several tens of nanoseconds. Figure 5(a) and 5(b) show the fluorescence dynamics of the Eu^{3+} ions in different time scales. It was obvious that the fluorescence intensity of Eu^{3+} increased initially, then decayed with a long exponential tail. There exist two increasing time constants, one constant was 3.2 ± 0.2 μs and the other one was smaller than 0.5 μs. The two increasing time constants of Eu^{3+} ions were properly unity with the two decay constants of Eu^{2+} ions. The decay time constant of Eu^{3+} ions in the 2 wt % Eu-doped glass was
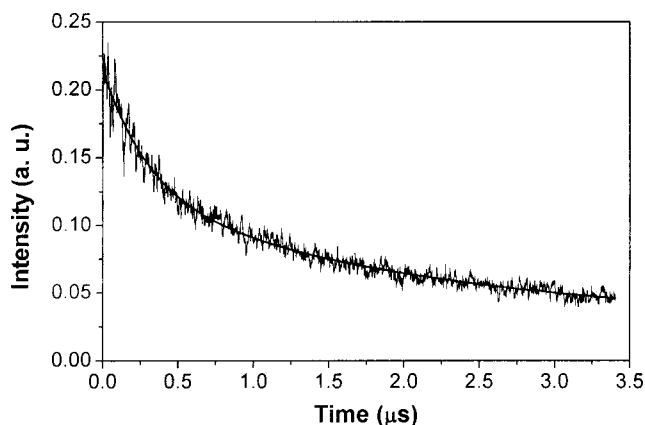

FIG. 4. Fluorescence dynamics of Eu^{2+} in the aluminosilicate glass. The excitation wavelength is 266 nm, the monitor position is 500 nm. The smooth lines are biexponential fitting functions.
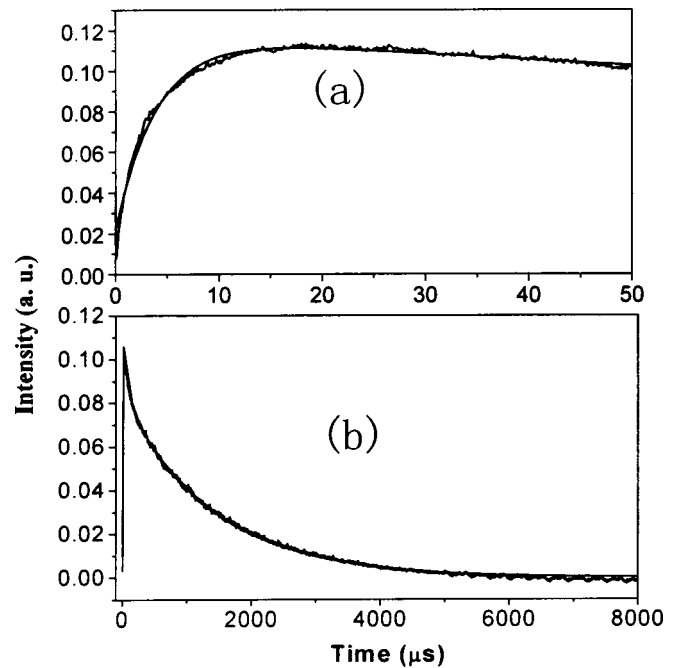

FIG. 5. Fluorescence dynamics of Eu^{3+} (a) in a short time scale and (b) in a long time scale. The excitation wavelength is 266 nm, the monitor position is 614 nm. The smooth lines are exponential fitting functions.

1500 ± 50 μs. The fluorescence intensity and the decay time constants of the glasses with different Eu concentration were measured and compared, which were listed in Table II. The fluorescence intensity increased as the Eu concentration increased. On the other hand, the decay time constant became slightly smaller. This result indicates that the concentration quenching was not strong as the Eu_2O_3 concentration was smaller that 2 wt % in the oxygen glasses.

Figure 6 shows the time-resolved spectra of the glass. It was more obvious that the emission increase of Eu^{3+} ions was accompanied by the fluorescence decrease of Eu^{2+} ions. The above results implied that the energy transfer occurred between Eu^{2+} and Eu^{3+} ions. Under the coexistence of the Eu^{2+} and Eu^{3+} ions, the excited Eu^{2+} in the $4f^65d$ level Eg nonradiatively relaxed to the ground state $^8S_{7/2}$, and the energy corresponding to the $4f^65d\rightarrow{}^8S_{7/2}$ transition caused excitation of the Eu^{3+} ion to the ^5D_J state.

C. Thermal population in $^7F_1$ and its effect on line shape

Figure 7 shows the resonant excitation spectra as different sites in the transition of $^5D_0-^7F_2$ were detected. The peak of $^7F_0-^5D_0$ was around 578 nm and had a small shift with monitor wavelength. It was interesting to observe that a shoulder appeared in the long wavelength side of $^7F_0-^5D_0$,

TABLE II. Variation of relative emission intensity and fluorescence lifetime of the $^5D_0-^7F_2$ transitions of Eu^{3+} ions on Eu concentration.

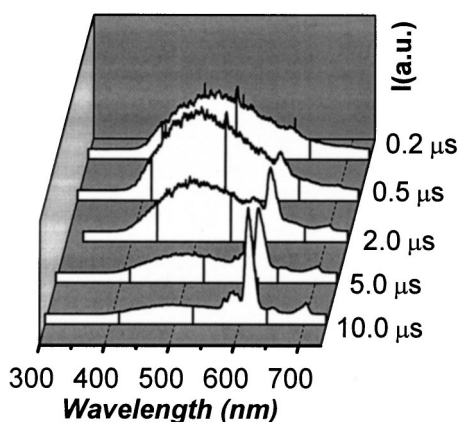| Eu concentration	0.5 wt. %	1 wt. %	2 wt. %
Relative Intensity (a.u.)	0.75	0.84	1.00
Lifetime (μs)	1800	1650	1500

FIG. 6. Time-resolution spectra of the Eu-doped glass. In the measurement, the excitation wavelength is 266 nm.

FIG. 8. The emission spectra of the $^5D_0-^7F_2$ transition while selectively excite the transition of $^7F_0-^5D_0$.

which originated from the transition of $^7F_1-^5D_0$. In fact, the average energy separation between $^7F_0$ and $^7F_1$ levels was only ~300 cm^{-1}. At room temperature, a great number of electrons on $^7F_0$ were thermally excited to $^7F_1$. The population of thermally excited electrons from $^7F_0$ to $^7F_1$, N_1, is proportional to $N_0 \exp(-E_{10}/k_BT)$, where N_0 is the electron population of $^7F_0$, E_{10} is the energy separation between $^7F_1$ and $^7F_0$, and k_B is the Boltsmann constant; T is the absolute temperature. According to the equation, N_1/N_0 was determined to be as large as ~60%. The excitation intensity of the $^7F_1-^5D_0$ transition was much smaller than that of $^7F_0-^5D_0$ due to the small excitation cross section of the $^7F_1-^5D_0$ transition. Based on the excitation spectra, we estimate the transition probability of $^7F_0-^5D_0$ was about 8–10 times larger than that of $^7F_1-^5D_0$.

Figure 8 shows the selective excitation spectra of the $^7F_0-^5D_0$ transition monitored by the fluorescence of $^5D_0-^7F_2$. (Spectra were measured with a resolution of 2.5 nm.) It was obvious that the line shape of $^5D_0-^7F_2$, varied significantly with the excitation wavelength. At 10 K, we measured the selective excitation spectra under the excitation of the narrower dye laser. Unfortunately, the Stark splitting peaks were not distinguished ($^7F_2$ configuration should have five components considering the Stark effect). The spectral shape almost did not vary with the excitation wavelength. (The line shape measured at 10 K and with high resolution is as similar as that measured with the excitation of 578 and 266 nm lights, as shown in Figs. 2 and 8.) This indicates that
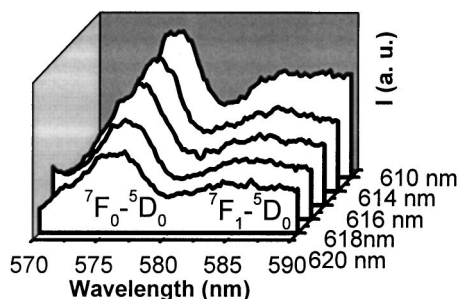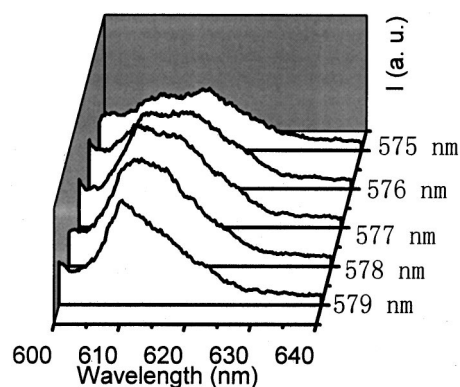
the variation of the $^5D_0-^7F_2$ line shape with excitation wavelength at room temperature is not caused by the crystal splitting effect, but rather by thermal population of the $^7F_1$ state. In fact, both the electrons in the $^7F_0$ and $^7F_1$ states could be optically excited into the $^5D_0$ state, and then radiatively relaxed to the $^7F_2$ one, leading to the configuration change of the $^5D_0-^7F_2$ transition.

D. Fluorescence change before and after light irradiation

Figure 9 shows the resonant excitation spectra of the $^7F_0-^5D_0$ transition before and after laser irradiation. After
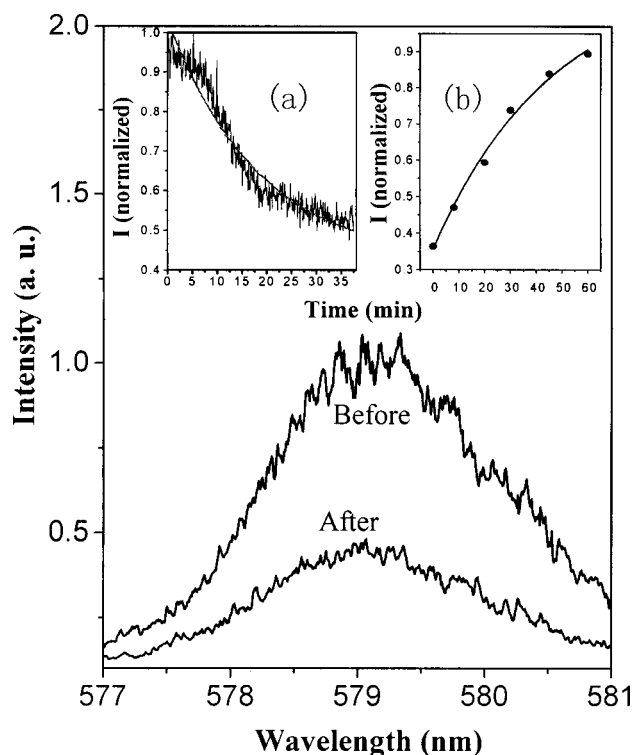
FIG. 9. Excitation spectra of $^7F_0-^5D_0$ measured before and after laser irradiation with a resonance light within the $^7F_0-^5D_0$ transition (579 nm). The spectra were obtained by monitoring the $^5D_0-^7F_2$ emission at 614 nm. (Inset) Dependence of the $^7F_0-^5D_0$ excitation line intensity on (a) irradiation time and (b) waiting time while the irradiation light was shut off.

FIG. 7. Resonance excitation spectra while different sites within the $^5D_0-^7F_2$ transition were monitored.

FIG. 10. Fluorescence spectra of the Eu-doped glass excited by a 375 nm light: (a) before irradiation, (b) after irradiation, and (c) difference spectrum. (Inset) The fluorescence intensity of Eu^{2+} as a function of irradiation time.
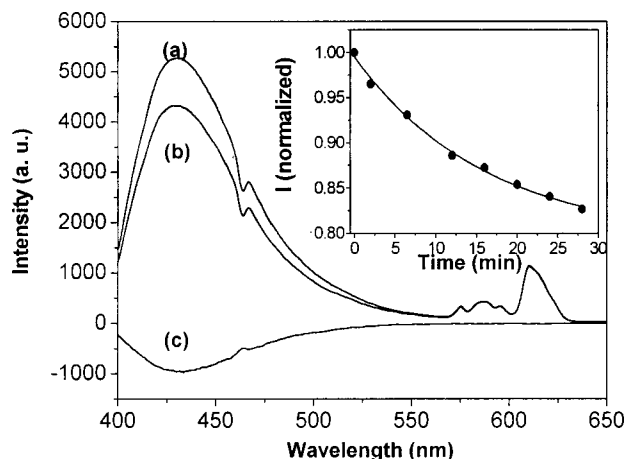
being irradiated by a laser light with a power density of ~20 mW/mm^2 for 20 min, corresponding to the transition of $^7F_0 - {}^5D_0$, no hole was formed even at 10 K. In fact, the excitation intensity of the $^7F_0 - {}^5D_0$ transition decreased. This behavior was quite different from the other Eu^{3+}-doped aluminosilicate glasses, which could be hole burned at low temperature.[10,21] The intensity decrease of the $^7F_0 - {}^5D_0$ transition was also observed at room temperature, but the variation was smaller in comparison to the decrease at low temperature. As the irradiation light was shut off, the excitation intensity of $^7F_0 - {}^5D_0$ increased and tended to recover to its original state. The detailed mechanism of the fluorescence decrease is not clearly understood. One possible process is that Eu^{3+} ions capture electrons at nearby trap sites, which are then transferred into Eu^{2+} ions. However, no evidence indicated that Eu^{2+} ions increased after irradiation. On the contrary, the irradiation with a weak light at 578 nm resulted in a decrease in fluorescence of Eu^{2+}, which would be demonstrated later. The other possible process is the optically excited rearrangement of local ions/molecules surrounding the Eu^{3+} ions. The rearrangement of local ions/molecules caused the fluorescence of Eu^{3+} to decrease. In Sm^{2+} and Eu^{3+}-doped aluminosilicate glasses prepared by the sol–gel method, the hole formation was related to the optically activated rearrangement of OH bonds surrounding Eu^{3+} or Sm^{2+} ions.[22,23] Insets (a) and (b) show the irradiation dynamics and recovery dynamics of the intensity of the $^7F_0 - {}^5D_0$ excitation line, respectively. At 10 K, an exponential decay constant was determined to be 40 min. The other constant was much longer. At room temperature, the decay constant was also on the order of several tens of minutes. This indicated that the thermal activation energy between the unchanged state and the changed state were two kinds at least (or a continuous band). At low temperature, both the two changed states of Eu^{3+} ions could persistently exist. On the other hand, only the changed state with a deep thermal depth could persistently exist at room temperature.

Figure 10 shows the spectra before and after irradiation with a 375 nm light. It was evident that the fluorescence of

Eu^{2+} decreased. The fluorescence of the $^5D_0 - {}^7F_J$ transition of Eu^{3+} did not change. The inset in Fig. 10 shows the fluorescence intensity of Eu^{2+} ions as a function of irradiation time. The fluorescence intensity of Eu^{2+} ions can be adequately fit by an exponential function of time. It should be noted that the peak position of the fluorescence of Eu^{2+} ions varied depending on the excitation wavelength. The peak position of the fluorescence of Eu^{2+} shifted to the red side as the excitation wavelength decreased. The fluorescence spectra were also measured after irradiation with the lamp light separated at the other wavelengths, 266 and 578 nm, the latter wavelength corresponding to the $^7F_0 - {}^5D_0$ transition of Eu^{3+} ions. The results were the same as that shown in Fig. 10. The fluorescence of Eu^{2+} ions decreased while the fluorescence of Eu^{3+} did not change. The fluorescence of Eu-doped glass was also measured and compared before and after the x-ray irradiation for 20 min. The fluorescence of both Eu^{2+} and Eu^{3+} ions decreased after the irradiation. The structural change was persistent at room temperature. This could result from the energetic x ray, which optically excited the electrons from the valence band or some impurity levels into the conduction band. The electrons then relax from the conduction band to the other impurity levels and the valence band. These processes are complicated and thus may lead to the structural variation observed. The fluorescence ratio of Eu^{2+} to Eu^{3+} showed no change, therefore, it was difficult to judge if the valence transfer occurred between Eu^{3+} and Eu^{2+} ions. The local environment surrounding Eu^{2+}/Eu^{3+} ions and the photorefractive index were changed by x-ray irradiation.

E. Two-level system in the aluminosilicate glasses

In most of the rare earth doped crystallized materials and glasses, a spectral hole can be burned at low temperature. In crystallized systems, hole width is generally twice that of the homogeneous linewidth. In amorphous solids such as glasses and polymers, the homogeneous linewidth as well as hole width varies with aging time. The quasihomogeneous linewidth is called spectral diffusion. In glasses, spectral diffusion is believed to be caused by the interaction of impurities with two-level systems (TLSs) that exist in the glass network.[24–26] At present, the origin of TLS is not clear in any system. According to the theory of standard TLS, the homogeneous linewidth equals log(t). In our Eu-doped glasses, the fluorescence decrease of Eu^{3+} ions (while the glass was irradiated by a frequency-selective light) can also be attributed to spectral diffusion. For the glass compositions studied, extensive TLSs are expected. These TLS strongly interact with Eu^{3+} ions and lead to an increase in the homogeneous linewidth of Eu^{3+} ions with time. The hole width after extensive pumping (20 min) should be close to the inhomogeneous linewidth of Eu^{3+}, leading to fluorescence bleaching. At present, we also cannot identify the origin of TLS in our glass network.

IV. CONCLUSIONS

Eu-doped aluminosilicate glasses with strong emission of Eu^{2+} and Eu^{3+} ions were prepared. The fluorescence be-

havior of the glasses was systematically studied. It is concluded that a large population of electrons in the $^7F_0$ state was thermally excited into the $^7F_1$ state at room temperature. The electrons in the $^7F_1$ level were optically excited into the $^5D_0$ state and then radiatively relaxed to the $^7F_2$ state. Due to the diffusion of $^7F_1$ and $^7F_0$ states, the line shape of the $^5D_0-^7F_2$ transition varied with the excitation wavelength.

The time resolved spectra and the fluorescence dynamics indicated that the fluorescence increase of Eu^{3+} ions corresponded to the fluorescence decrease of Eu^{2+} ions. The energy transfer from Eu^{2+} ions to Eu^{3+} ions occurred through excitation of the Eu^{3+} ion to the ^5D_J level, by the $4f^65d$ to $^8S_{7/2}$ transition of the Eu^{2+} ion.

The fluorescence and excitation spectra were investigated before and after irradiation at different wavelengths. The fluorescence of Eu^{2+} decreased after the glass was irradiated with 266, 375, and 578 nm lights while the fluorescence of Eu^{3+} ions did not change. After being irradiated by the x ray, both the fluorescence of Eu^{2+} and Eu^{3+} decreased. The emission decrease of Eu^{2+} and Eu^{3+} ions (after light irradiation) was not caused by the valence transfer between Eu^{2+} and Eu^{3+} ions, but rather caused by the local environment rearrangement of Eu^{3+} and/or Eu^{2+} ions.

No spectral hole was formed by the selective irradiation with a narrow laser line, even at low temperature (10 K). In fact, the excitation intensity of $^7F_0-^5D_0$ decreased after hole burning. The results suggested that TLS extensively existed in the glass network and strongly interacted with the Eu^{3+} ions which resulted in spectral diffusion. Further work is required at different host composition to clarify the origin of TLS.

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