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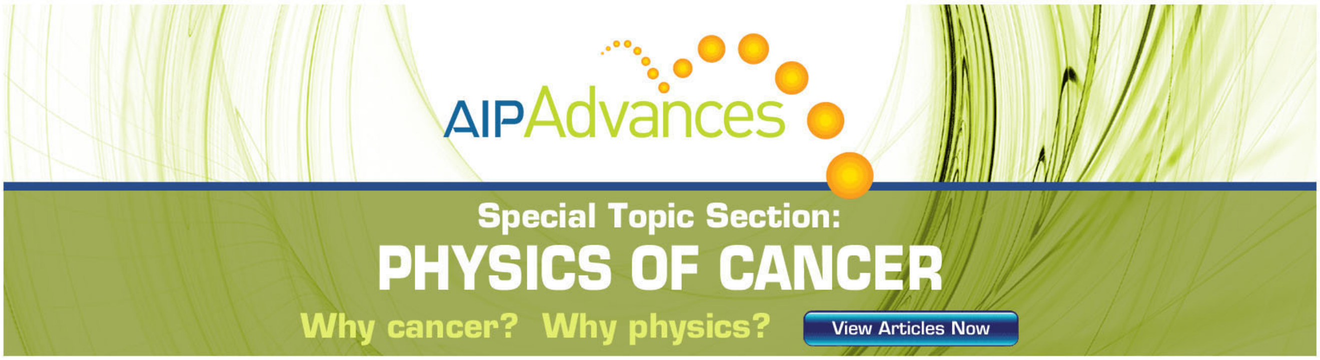

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Temperature dependence of the fluorescence of Eu^{3+}-ion doped in various silicate glasses

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The temperature dependence of emission intensity of the Eu^{3+} ion in several silicate glasses was investigated between 77 and 670 K, under the excitation of a 488-nm laser. It was observed that in some glasses the total emission intensity of the Eu^{3+} ion had a maximum at a certain temperature below 670 K, while in the other glasses increased monotonically with the increasing temperature. A unified theoretical model was given considering thermal activation, phonon-assisted absorption and nonradiative energy transfer processes. Based on the model, all of the experimental data were well fitted. The temperature-quenching channels were discussed, and the temperature-quenching rates were determined. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1476962]

I. INTRODUCTION

The rare-earth ions have attracted much attention, because they have large potential for applications in some optical fields, such as lasers, optical fibers, phosphors, electroluminescent devices, and high density optical storage.[1–7] The Eu^{3+} ion, of which the lowest excited level ($^5D_0$) of the $4f^6$ configuration is situated below the $4f^55d$ configuration. It shows very sharp emission lines extending from the visible to the near-infrared region. The persistent spectral hole burning (PSHB) can be performed within its $^7F_0-^5D_0$ transition.[8–10] As known, PSHB has potential application as a media of high density optical storage. For practical application, PSHB at room temperature is required. The first observation of PSHB at room temperature is in a Sm^{2+}-ion-doped fluoride mixed crystal.[11,12] Recently, PSHB phenomenon at roomtemperature has been observed in Eu^{3+}-doped as well as Sm^{2+}-doped glasses.[13–16] For this sake, Eu^{3+}-ion doped in glass hosts is attracting current interests.

The luminescent behavior of the Eu^{3+} ion in silicate glasses has been frequently studied.[17–19] However, the temperature-dependent luminescent behavior was seldom studied. In this article, we study the temperature-dependent characteristic of the emission of the Eu^{3+} ion under the pump of a 488-nm light. The understanding of the temperature-dependent luminescent process is helpful to search for new materials optical storage at high temperature and phosphors with high quantum efficiency.

II. EXPERIMENTS

Three kinds of oxide glasses normally containing 0.5–2 wt % Eu_2O_3 were prepared by melting. Compositions of the host glasses are $68.0SiO_2-9.4B_2O_3-7.4Na_2O-14.2K_2O_3$, $62SiO_2-20Al_2O_3-6Li_2O-2MgO-2Na_2O-2TiO_2-2ZrO_2-3P_2O_5-1Sb_2O_3$, and $50SiO_2-20Al_2O_3-15K_2O-15Na_2O$ in mol %. These glasses are named BSiO, GCO, and AlSiO, respectively. In the preparation, all of the starting materials (commercial available) were mixed together and stirred until the mixture became homogeneous, then put into a furnace in the air. After keeping at 1450 °C for 4 h and slowly cooling down to room temperature, the colorless transparent glasses were formed. These glasses were cut into small pieces (1 cm×1 cm) with thickness of 1 mm and were well polished.

In the measurements, the samples were put into a liquid nitrogen cycling system, in which the temperature varied from 77 to 670 K. A continuous 488-nm light came from an argon laser was used as excitation. The fluorescence was measured by a UV-Lab Raman Infinity (made by Jobin–Yvon Company) with resolution of 2 cm^{-1}.

III. RESULTS AND DISCUSSION

A. Dependence of the emission intensity of the Eu^{3+} on temperature

Figures 1(a) and 1(b) show, respectively, the emission spectra of the Eu^{3+} ions at various temperatures in BSiO: Eu (1.5%) and AlSiO: Eu (1%). Strong emissions due to the $^5D_0-^7F_J$ transition is observed in both the two glasses. The $^5D_1-^7F_J$ emission intensity is two orders smaller than that of the $^5D_0-^7F_J$ one, indicating the nonradiative rate of the $^5D_1-^5D_0$ transition is much larger than the radiative rate of the $^5D_1-^7F_J$ one. Therefore, the $\Sigma_J\ ^5D_0-^7F_J$ emission intensity represents the total emission intensity of the Eu^{3+} ion. It is obvious that the emission intensity of the Eu^{3+} ion varies greatly with temperature, and the variation in the two hosts shows a remarkable difference. On one hand, the emission intensity of the Eu^{3+} ion in the host of BSiO: Eu (1.5%) increases monotonously as the temperature increases. On the

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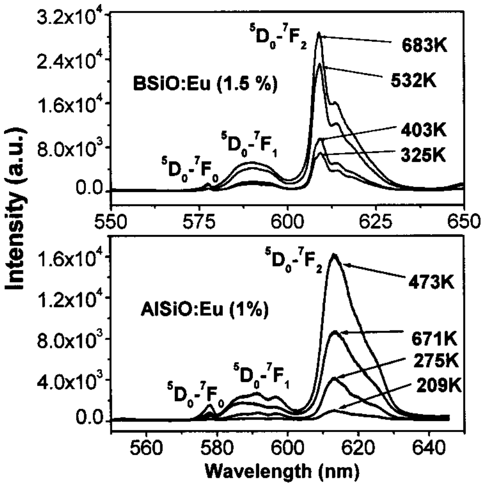

FIG. 1. Emission spectra of the $^5D_0-^7F_J$ (J=0, 1, 2) transitions of the Eu^{3+} ion in BSiO: Eu (1.5%) and AlSiO: Eu (1%) glasses at various temperatures.

other hand, the intensity of the Eu^{3+} ion in AlSiO: Eu (1.0%) increases with temperature initially and approaches at a maximum around 473 K, then it decreases as temperature increases continuously. Actually, we also measured the temperature dependence of the emissions of the Eu^{3+} ion in the other glasses. Figure 2 shows the temperature dependence of the emission intensity of the Eu^{3+} ion in all the glasses. As shown, the emission intensity of the Eu^{3+} ion in BSiO glasses both increases monotonously with temperature, and the intensity of the Eu^{3+} ion in GCO glasses both has a maximum. For the AlSiO glasses, the intensity in the glass doped with 1.0% of Eu_2O_3 has a maximum, while that in the glass doped with 0.5% of Eu_2O_3 increases monotonously with temperature. In one word, the variation of the emission intensity of the Eu^{3+} ion is dependent of not only the composition of the host, but also the concentration of Eu^{3+} ion.

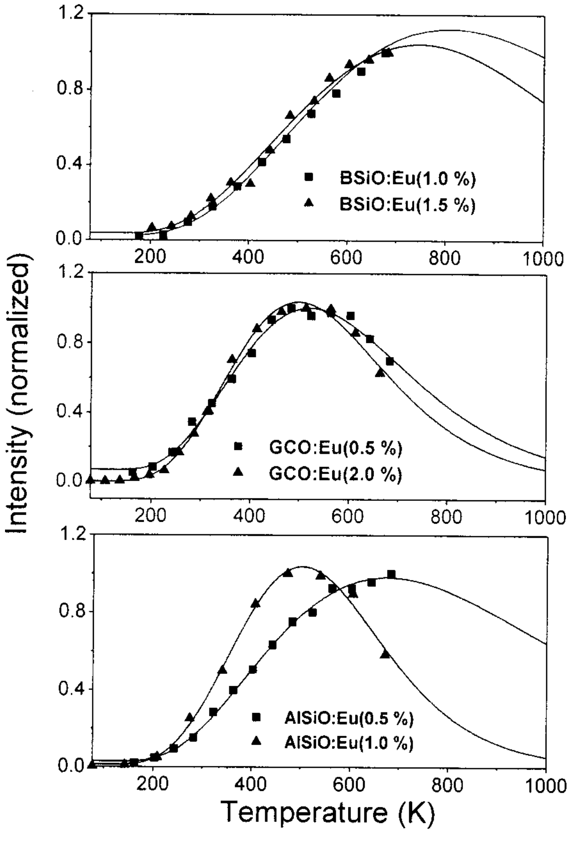

FIG. 2. Normalized emission intensity of $\Sigma_J\ ^5D_0-^7F_J$ as a function of temperature, The dots are experimental data and the solid lines are fitting functions.

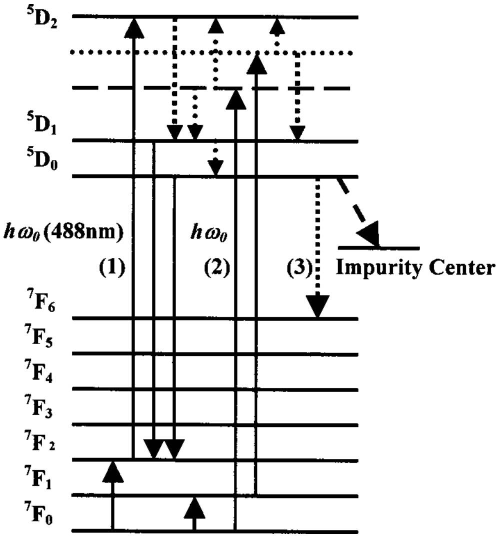

FIG. 3. Schematic of the luminescence processes under the excitation of the 488-nm light. The solid lines present radiative transitions and the dashed lines are nonradiative ones.

To explain the behavior of temperature-dependent luminescence, the scheme of the energy levels of the Eu^{3+} ion and the corresponding transition processes under the excitation of the 488-nm light was drawn as Fig. 3. The energy of a 488-nm photon (20491 cm^{-1}) is lower than the absorption limit of the host. Under the excitation of the 488-nm light, there exist two paths to excite electrons on ^7F_J levels to the excited states. On one hand, the electrons on $^7F_0$ can be thermally activated to the closed ^7F_J states, $^7F_1$, and $^7F_2$. Taking into account the $^7F_2$ splitting and the widths of corresponding Stark levels, the 488-nm excitation can be in resonance with $^7F_2-^5D_{2,}$ (see channel 1 in Fig. 3). Here, we neglected the other possible transitions from the higher ^7F_J states (J=3, 4, 5, and 6), because the thermal populations of electron in these states are negligible compared to the ^7F_J (J=0, 1, 2) states (see Fig. 4). On the other hand, the electrons in $^7F_0$ and/or $^7F_1$ can be nonresonantly excited to $^5D_1$ and $^5D_2$ levels with the assistant of phonons (see channel 2 in Fig. 3). As the temperature is high enough, phonon-assisted absorption occurs. These two processes may cause the emission intensity of the Eu^{3+} ion to increase as the temperature increases.

Actually, the temperature-quenching effect becomes strong as the temperature is elevated, which tends to cause the emission intensity of Eu^{3+} ion to decrease. The temperature-quenching channels include the nonradiative transitions from $^5D_0$ to each ^7F_J level and the energy transfer from one excited Eu^{3+} ion to the other Eu^{3+} ions or to the other centers nearby (see channel 3 in Fig. 3). Here, we do not discuss the possible energy transfer processes from $^5D_2$

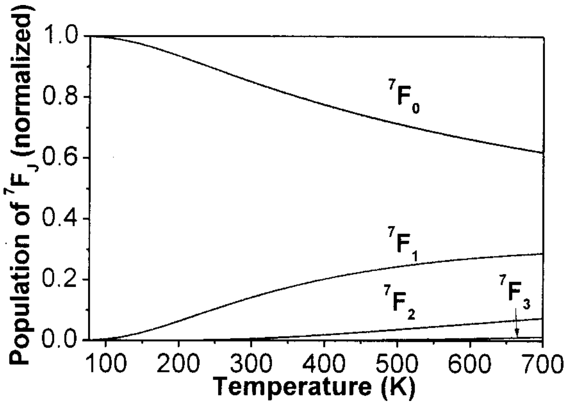

FIG. 4. Normalized population of ^7F_J as a function of temperature calculated by $N_J(T) = e^{-\Delta E_J/kT}/\Sigma_J e^{-\Delta E_J/kT}$, $J=0$, 1, 2, 3.

and $^5D_1$, because we consider that the nonradiative transition rates of $^5D_2-^5D_1$ and $^5D_1-^5D_0$ are much larger than the possible energy transfer rate. This can be concluded from the fact that the emission intensity of the $^5D_0-^7F_J$ transition is dominant at any temperature. As the contribution of the temperature-quenching effect is dominant in comparison with that of the thermally excited emissions, the emission intensity of the Eu^{3+} ion should decrease. This has explained why the emission intensity of the Eu^{3+} ions in some glasses has a maximum under the excitation of the 488-nm light.

B. Luminescent dynamic equations

The emission processes of the Eu^{3+} ions under the excitation of the 488-nm light was theoretically considered base on the luminescent dynamic equations. In the light of the emission spectra, it was concluded that $W_{21} \gg \gamma_2$, and $W_{10} \gg \gamma_1$ where W_{21}, W_{10}, γ_2, and γ_1 are, respectively, the nonradiative transition rates of $^5D_2-^5D_1$ and $^5D_1-^5D_0$, and the radiative transition rates of $\Sigma_J\,^5D_2-^7F_J$ and $\Sigma_J\,^5D_1-^7F_J$. Therefore, the emission intensity of Σ_J $^5D_0-^7F_J$, $I(T)$, can be written approximately as,

$$I(T) \propto \frac{\Sigma\, I N_J(T)\sigma_J(T)}{1+W_T(T)/\gamma_0}, \quad J=0,\ 1,\ 2, \tag{1}$$

where I is the power density of the pumping light, W_T is the temperature-quenching rate, γ_0 is the radiative transition rate of $\Sigma_J\,^5D_0-^7F_J$, N_J is the electron population on ^7F_J, $\sigma_J(T)$ is the total absorption cross section from ^7F_J to $^5D_2$ and $^5D_1$ (J=0, 1, and 2). The normalized thermal population on ^7F_J, can be expressed as,

$$N_J(T) = \frac{e^{-\Delta E_J/kT}}{\Sigma_J e^{-\Delta E_J/kT}}, \quad J=0,\ 1,\ 2, \tag{2}$$

where ΔE_J is the energy separation from ^7F_J to $^7F_0$, k is Boltzmann's constant. As is well known, the multiphonon relaxation rate in the rare-earth ions for emitting and absorbing phonon processes can be, respectively, written as[20]

$$\begin{aligned} W_{NR} &= Ce^{-\alpha\Delta E}\times[1+<n>]^{\Delta E/h\omega} \quad \text{emitting phonons} \\ &= Ce^{-\alpha\Delta E}\times<n>^{\Delta E/h\omega} \quad \text{absorbing phonons} \end{aligned} \tag{3}$$

with

$$\alpha = \frac{1}{h\omega}[\ln(\Delta E/h\omega)-1-\ln s], \tag{4}$$

where C is a constant dependent of host and almost independent of temperature, s is Huang–Rhys factor, $\langle n \rangle = 1/\exp(h\omega/kT)$-1 is the phonon density, ΔE is the energy difference of the two levels, between which the multiphonon relaxation happens. Based on Eqs. (3) and (4), the phonon-assisted absorption cross sections from $^7F_0$ to $^5D_2$ and $^5D_1$ and from $^7F_1$ to $^5D_2$ and $^5D_1$, were written, respectively, as

$$\begin{aligned} \sigma_0(T) &= \sigma_A[e^{-\alpha E_{01}}(1+\langle n \rangle)^{E_{01}/h\omega}+e^{-\alpha E_{02}}\langle n \rangle^{E_{02}/h\omega}] \\ \sigma_1(T) &= \sigma_A[e^{-\alpha E_{11}}(1+\langle n \rangle)^{E_{11}/h\omega}+e^{-\alpha E_{12}}\langle n \rangle^{E_{12}/h\omega}], \end{aligned} \tag{5}$$

where σ_A is a constant, E_{01} *and* E_{02} are energy differences between the energy of a 488-nm photon and the energy separation from $^7F_0$ to the $^5D_1$ and $^5D_2$ levels, E_{11} *and* E_{12} are energy differences between the energy of a 488-nm photon and the energy separation from $^7F_1$ to the $^5D_1$ and $^5D_2$ levels, respectively, $h\omega$ is the phonon energy. In Eq. (1), σ_2 is approximately equal to the resonant absorption cross section of $^7F_2-^5D_2$. It is independent of temperature. Based on the temperature dependence of the emissions of the Eu^{3+} ion under the excitation of a resonant 325-nm light in the glasses, the temperature-quenching rate is experimentally determined as[21]

$$W_T = W_T(0)e^{T/T_C}, \tag{6}$$

where $W_T(0)$ is the temperature-quenching rate at 0 K, T_c is a temperature constant. According to Eqs. (1)–(4), the emission intensity of the Eu^{3+} ion as a function of temperature can be rewritten as

$$\begin{aligned} I(T) \approx{} & \frac{\beta_1\times[e^{-\alpha E_{01}}(1+\langle n \rangle)^{E_{01}/h\omega}+e^{-\alpha E_{02}}\langle n \rangle^{E_{02}/h\omega}]}{(1+e^{-\Delta E_1/kT}+e^{-\Delta E_2/kT})\times(1+\beta_3 e^{T/T_C})} + \frac{\beta_1 e^{-\Delta E_1/kT}\times[e^{-\alpha E_{11}}(1+\langle n \rangle)^{E_{11}/h\omega}+e^{-\alpha E_{12}}\langle n \rangle^{E_{12}/h\omega}]}{(1+e^{-\Delta E_1/kT}+e^{-\Delta E_2/kT})\times(1+\beta_3 e^{T/T_C})} \\ & + \frac{\beta_2 e^{-\Delta E_2/kT}}{(1+e^{-\Delta E_1/kT}+e^{-\Delta E_2/kT})\times(1+\beta_3 e^{T/T_C})}, \end{aligned} \tag{7}$$

where $\beta_1 = I\sigma_1$, $\beta_2 = I\sigma_2$, and $\beta_3 = W_T(0)/\gamma_0$.

TABLE I. A list of parameters β_1, β_2, β_3, and T_C (K) in various glasses.

Silicate glasses	β_1	β_2	β_3	T_C (K)
BSiO: Eu (1.0%)	3.6	18.6	4.1×10^{-2}	288
BSiO: Eu (1.5%)	7.6	19.1	1.8×10^{-2}	200
GCO: Eu (0.5%)	15.6	63.3	4.5×10^{-2}	140
GCO: Eu (2.0%)	0.37	70.8	2.1×10^{-2}	116
AlSiO: Eu (0.5%)	8.1	45.3	2.0×10^{-1}	269
AlSiO: Eu (1.0%)	2.8	57.4	9.1×10^{-3}	104

C. Fitting results

Using Eq. (7), we well fitted the experimental data. The fitting functions were shown in Fig. 2. In the fitting, we chose $h\omega=1080\ \mathrm{cm}^{-1}$ (we measured the Raman spectra of all the glasses and observed the Raman peaks at $1080\ \mathrm{cm}^{-1}$ and $600\ \mathrm{cm}^{-1}$, which originated from the Si—O—Si and the Al—O^- bonds, respectively.[22] The $1080\ \mathrm{cm}^{-1}$ peak is dominant for all glasses), $\triangle E_1=372\ \mathrm{cm}^{-1}$, $\triangle E_2=1026\ \mathrm{cm}^{-1}$, $E_{01}=1464\ \mathrm{cm}^{-1}$, $E_{02}=992\ \mathrm{cm}^{-1}$, $E_{11}=1836\ \mathrm{cm}^{-1}$, $E_{12}=620\ \mathrm{cm}^{-1}$, and $s=0.01$ (In rare-earth ions, the value of s is between 0.01–0.1. In inorganic hosts, s is smaller than that in organic ones, therefore, s is chosen to be 0.01). It should also be noted that, the energy levels of the Eu^{3+} ion in LaCl_3 are used to calculate the energy differences above, because the energies of the $f-f$ transitions of the Eu^{3+} ions have only a little variation (several ten wave numbers) in different hosts. By fitting, the parameters β_1, β_2, β_3, and T_C were determined, as shown in Table I.

As shown in Fig. 2, the maximum of luminescent intensity of Eu^{3+} ion appears at 492 K for GCO: Eu (2.0%), at 527 K for GCO: Eu (0.5%) and at 486 K for AlSiO: Eu (1.0%). Actually, as the temperature is high enough, the intensity of Eu^{3+} ion in BSiO and AlSiO: Eu (0.5%) also has a maximum. According to the fitting results, the maximums should appear at 695 K for AlSiO: Eu (0.5%), at 804 K for BSiO: Eu (1.0%), and at 741 K for BSiO: Eu (1.5%).

In Table I, the value of parameter β_3 is much less 1, for all the glasses. This is in consistent with the fact that the radiative transition rate of ${}^5D_0-{}^7F_J$ is much larger than the temperature-quenching rate, $W_T(0)$. Figure 5 shows the calculated emission intensity contributed by the excitation of ${}^7F_0-{}^5D_1$ and ${}^5D_2$ and ${}^7F_1-{}^5D_1$ and ${}^5D_2$ and ${}^7F_2-{}^5D_2$ transitions. As the temperature is lower, the phonon-assisted excitation from ${}^7F_0$ to ${}^5D_1$ and ${}^5D_2$ is dominant. The resonant excitation of ${}^7F_2-{}^5D_2$ and the phonon-assisted excitation from ${}^7F_1$ to ${}^5D_1$ and ${}^5D_2$ are negligible due to the lower electron populations on ${}^7F_1$ and ${}^7F_2$. As the temperature increases, the population on ${}^7F_2$ becomes more and more, thus the resonant excitation of ${}^7F_2-{}^5D_2$ is dominant.

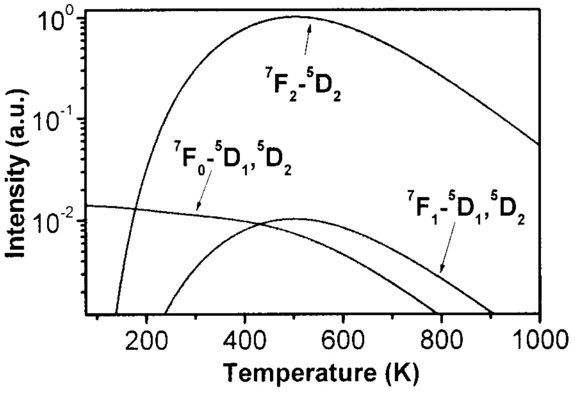

FIG. 5. Emission intensity contributed by the excitation of ${}^7F_0-{}^5D_1$, ${}^5D_2$ and ${}^7F_1-{}^5D_1$, ${}^5D_2$ and ${}^7F_2-{}^5D_2$ transitions calculated according to Eq. (7). In the calculation, the parameters are $\beta_1=2.8$, $\beta_2=57.4$, $\beta_3=9.1\times10^{-3}$, and $T_C\ (\mathrm{K})=104$ K.

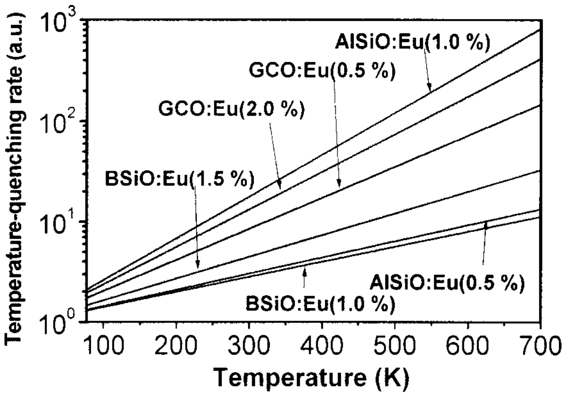

FIG. 6. Temperature-quenching rate as a function of temperature in various silicate glasses calculated by $W_T=W_T(0)e^{T/T_C}$, The value of T_C are 288 K (BSiO: Eu 1.0%), 200 K (BSiO: Eu 1.5%), 140 K (GCO: Eu 0.5%), 116 K (GCO: Eu 2.0%), 269 K (AlSiO: Eu 0.5%), and 104 K (AlSiO: Eu 1.0%), respectively.

D. Temperature-quenching effect

Base on the values of T_C listed in Table I, the temperature-quenching rate as a function of temperature was drawn as Fig. 6. It is clear that the temperature-quenching rate depends on the host composition and the concentration of the Eu^{3+} ion. The energy separation from ${}^5D_0$ to the highest ${}^7F_J$ level $(J=6)$ is $\sim 12\,400\ \mathrm{cm}^{-1}$. If an electron nonradiatively relaxes from the ${}^5D_0$ to the ${}^7F_J$ level, at least ten phonons will be released into the glass network. Considering only this nonradiative relaxation channel, the temperature-quenching rate will vary 3–4 times from 77 to 670 K. In some glasses, the real variation of the temperature-quenching rate is much larger than several times. suggesting that the other channel exists and contributes dominantly to the temperature-quenching effect. It is attributed to the energy transfer from the ${}^5D_0$ levels to the other Eu^{3+} ions or the other impurity centers. In BSiO and AlSiO glasses, the temperature-quenching rate obviously increases as the concentration of the Eu^{3+} ion increases. This suggests that the nonradiative relaxation process from ${}^5D_0$ to ${}^7F_J$ contributes dominantly to the temperature-quenching effect as the concentration of the Eu^{3+} ion is lower. On the other hand, the energy transfer process from one excited Eu^{3+} to the other Eu^{3+} ions contributes dominantly to the temperature-quenching effect as the concentration of Eu^{3+} ion is higher. In GCO glasses, the temperature-quenching rate has small variation with the concentration of the Eu^{3+} ion, suggesting that the energy may transfer from the Eu^{3+} ion to the other impurity centers nearby in the glass network.

IV. CONCLUSIONS

The temperature-dependent behavior of the emission intensity of the Eu^{3+} ion was studied in various silicate glasses, under the pump of a nonresonant 488-nm laser. In different glasses, the temperature dependence of the emissions of the Eu^{3+} ion showed an obvious difference. It was well explained by the same luminescent dynamic equations. In fact, the variation of the emission intensity of Eu^{3+} can be mainly attributed to two factors, the thermally excited emission from ${}^7F_2$ to ${}^5D_2$ and the temperature-quenching effect. The former tends to cause the intensity of Eu^{3+} increase with temperature, while the later tends to cause the intensity decrease. In some glasses such as BSiO: Eu and AlSiO: Eu (0.5%), the thermal excited emission of ${}^7F_2 - {}^5D_2$ was dominant all the time in the studied temperature range (77–670 K), thus the emission intensity of Eu^{3+} increased remarkably with temperature. In the other glasses such as GCO: Eu and AlSiO: Eu (1.0%), the temperature-quenching effect is comparable with the thermal excited emission of ${}^7F_2 - {}^5D_2$ when the glass is elevated to one certain temperature, thus a maximum of emission intensity occurs. In fact, as long as the temperature is high enough, the emission intensity of the Eu^{3+} ion will decrease finally in all the glasses. In all the glasses, the phonon-assisted transition rates from ${}^7F_0$ to ${}^5D_1$ and ${}^5D_2$ and from ${}^7F_1$ to ${}^5D_1$ and ${}^5D_2$ are much smaller than the resonant transition rate of ${}^7F_2 - {}^5D_2$.

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