Excitability of high-energy ultraviolet radiation for Dy³⁺ in antimony phosphate glasses

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 Dy^{3+} doped antimony phosphate (ZASP) glasses are synthesized and the specificity of the luminescence behavior is demonstrated. Different from the conventional long-wave ultraviolet (UVA) exciting cases, the excitable area of Dy^{3+} doped ZASP glasses is extended to high-energy ultraviolet radiation including medium-wave ultraviolet (UVB) and short-wave ultraviolet (UVC) spectral regions. The quantum efficiency for ${}^{4}F_{9/2}$ level of Dy^{3+} in low- and medium-concentration Dy_2O_3 doping cases reaches 95.0 % and 66.7 %, respectively, confirming the emission effectiveness from Dy^{3+} in ZASP glasses. The values of energy-transfer probability (P) have obvious difference while using 340 nm and 540 nm as monitoring wavelengths, so as the energy-transfer efficiencies (η), which are related to the energy-transfer processes from discrepant Sb³⁺ donors to Dy^{3+} acceptors, were in-equivalent. The effective excitability of high-energy ultraviolet radiation illustrates that Dy^{3+} doped ZASP glasses are a promising candidate in developing visible light sources, display devices and tunable visible lasers.

Keywords: Dy^{3+} ; antimony phosphate glasses; excitability of high-energy ultraviolet radiation; energy transfer

1. Introduction

There is a growing interest in the development of rare earth (RE^{3+}) doped glasses as they exhibit intense luminescence in the visible region for the use in high-density optical storage, luminescent sensor, undersea communication and medical diagnostics [1-7]. The host material of glass is a non-negligible factor for the active RE^{3+} to obtain efficient florescence [8–19]. Among all oxide glass laser materials, phosphate glasses have been a subject of considerable interest due to their unique properties such as high transparency, low production cost, high RE³⁺ ion solubility and large emission cross section [20-35]. Up to now, many researchers have devoted themselves with great enthusiasms to the studies of RE^{3+} doped phosphate glass systems, and great achievements have been reported on their spectral characteristics [36–38].

Among RE^{3+} ions, dysprosium ion (Dy^{3+}) in oxide glasses has narrow intense emissions at blue $({}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2})$ and yellow $({}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2})$ regions, and exhibits warm yellowish-white light to

naked eyes [39–44]. However, the effective excitation region of Dy^{3+} is restricted to the long-wave ultraviolet (UVA) area, showing the insufficiency in medium-wave ultraviolet (UVB) and short-wave ultraviolet (UVC) regions [45, 46]. Fortunately, antimony ion (Sb³⁺) is regarded as a promising candidate owing to low cost, extensive emission region and broad excitation zone covering the UVB and UVC regions. The introduction of Sb³⁺ will compensate effective excitation region of Dy^{3+} in highenergy ultraviolet region under a condition of possible energy transfer from Sb³⁺ to Dy^{3+} [47].

In this work, Dy^{3+} doped zinc aluminum antimony phosphate (ZASP) glasses have been fabricated and characterized, and the excitation band of Dy^{3+} is extended to high-energy UVB and UVC regions by the introduction of Sb^{3+} . Efficient energy transfer from Sb^{3+} to Dy^{3+} is assured and higher radiative transition parameters indicate that intense blue and yellow emissions are likely to be expected in ZASP glasses. Excitability of highenergy ultraviolet radiation may provide a broadened application of the glasses in making visible light sources.

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2. Experimental

2.1. Material synthesis

ZASP host glasses were prepared from Na₂CO₃, BaCO₃, ZnO, Al(OH)₃ and Sb₂O₃ powders and H₃PO₄ solution according to the molar composition 17.17 % Na₂O-0.97 % BaO-15.61 % ZnO-11.06 % Al₂O₃-5.66 % Sb₂O₃-49.53 % P2O5. Additional 0.26 %, 1.89 %, 3.09 % and 8.37 % of Dy₂O₃ were introduced into the ZASP glass composition to prepare Dy3+ doped ZASP glasses with different compositions. The wellmixed powders were first dropped with H₃PO₄ solution in an alumina crucible, then melted at 1200 °C for 50 min, and finally quenched to room temperature in a preheated graphite mould. The obtained glasses were subsequently annealed at 480 °C for 30 min, after that cooled down slowly to room temperature. For optical measurements, the annealed glasses were sliced and polished into pieces with parallel sides.

2.2. Measurements

The density of the 8.37 % Dy_2O_3 doped ZASP glasses, measured using Archimedes method, was $3.02 \text{ g} \cdot \text{cm}^{-3}$. The refractive indices, determined using a Metricon 2010 prism coupler, were 1.5717 at 635.96 nm and 1.5555 at 1546.9 nm, respectively. At all other wavelengths, the refractive indices can be derived by the Cauchy equation:

$$n = A + B/\lambda^2 \tag{1}$$

with A = 1.5522 nm^2 and B = 7884.7 nm². A differential thermal analysis (DTA) scan of the Dy³⁺ doped ZASP glasses was carried out by a WCR-2D differential thermal analyzer at a rate of 10 °C·min⁻¹ from room temperature to 800 °C. Amorphous structure of the glasses was confirmed by X-ray powder diffraction analysis using a D/Max-3B X-ray diffractometer at 30 kV and 30 mA. Excitation and emission spectra were determined by a Hitachi F-7000 fluorescence spectrophotometer and absorption spectrum was recorded by a PerkinElmer UV-Vis-NIR Lambda 950 double-beam spectrometer. Fluorescence decay curves were measured by a Jobin Yvon

Fluorolog-3 spectrophotometer equipped with an R928 photomultiplier tube and a flash Xe-lamp. Luminescence pictures were taken by a Sony DSLR-A200 digital camera. All the measurements were carried out at room temperature.

3. Results and discussion

3.1. Thermal properties of ZASP glasses

The XRD pattern of Dy^{3+} doped ZASP glass powder exhibits a broad diffraction peak characteristic of amorphous structure as shown in Fig. 1. The thermodynamic property of Dy^{3+} doped ZASP glass sample is presented by DTA curve in the inset of Fig. 1. Transition temperature (Tg) is measured to be 402 °C, and no crystallization onset point (Tx) is observed below the temperature of 570 °C. Thus, the thermal stability range ($\Delta T =$ Tx – Tg) is calculated to be more than 150 °C, which is broader than that in germanate glasses (67 °C) [48] and phosphate glasses (~100 °C) [49], indicating that Dy³⁺ doped ZASP glasses have better anti-crystallization ability in a wide temperature range, which is beneficial to drawing glass fiber.



Fig. 1. XRD pattern of 1.89 % Dy_2O_3 doped ZASP glass powder. Inset: DTA curve of related sample.

3.2. Broadband fluorescence of Sb³⁺ in ZASP glasses

Under different excitation wavelengths, ZASP host glasses exhibit diverse emission bands, which



Fig. 2. (a) Emission spectra of ZASP host glasses under different excitation wavelengths; (b) Gaussian fitting of the emission spectrum excited by 280 nm.

is represented in peak intensity, peak position and full-width at half-maximum (FWHM), as presented in Fig. 2a. When excitation wavelength is changed from 240 nm to 280 nm, emission intensity and FWHM increase, and emission peak blue-shifts from 445 nm to 416 nm. On the contrary, while excitation wavelength is changed from 280 nm to 315 nm, emission intensity and FWHM decrease and emission peak red-shifts from 416 nm to 440 nm.

Transition emission of Sb³⁺ relating to energylevel gap is sensitive to the coordinated surroundings, since Sb^{3+} possesses an electron in the outermost shell in both the ground state $(5s^2)$ and the excited state $(5s^{1}5p^{1})$ [50–52]. Previous studies have demonstrated that the shifting of emission peak is due to multiple luminescent centers located at multi-site positions [53–56]. The ZASP glasses are considered to contain [PO₄], [AlO₄], [AlO₆], [ZnO₄] and [ZnO₆] structural units, which provide different coordination fields with respective crystal field strengths. Usually, the stronger the surrounding crystal field strength the longer the emission wavelength. The ZASP glasses have various surroundings for emission centers providing a distribution of energetically different emission centers which allows their excitation wavelength selection [57]. Under the excitation of different wavelengths, all the emission bands of Sb^{3+} can be fitted as two Gaussian waves, which imply that ZASP glasses have two types of luminescence centers. Typically, when excited by 280 nm, the emission band of Sb³⁺ is decomposed into two waves peaking at 450 nm (22222 cm⁻¹) and 386 nm (25907 cm⁻¹), as presented in Fig. 2b. Energy-level diagram of differentiation of the luminescence centers Sb³⁺ (I) and Sb³⁺ (II) is illustrated in Fig. 3.



Fig. 3. Energy-level diagram of differentiation of luminescence centers Sb³⁺ (I) and Sb³⁺ (II) under 280 nm excitation.

Dissimilarity in excitation bands for different wavelength emissions originating from ${}^{1}S_{0} \rightarrow {}^{3}P_{1}$ transition of Sb³⁺ centers has been observed under superposition effect of respective Sb³⁺ luminescence centers, as illustrated on the left in Fig. 4. For 340 nm and 540 nm emissions, sensitive

excitation regions are 255 nm to 287 nm and 227 nm to 289 nm, respectively. The experimental lifetime:

$$\tau = \int_0^\infty t I(t) dt \bigg/ \int_0^\infty I(t) dt \tag{2}$$

for 340 nm ($\tau_{Sb-340 \text{ nm}}$) and 540 nm ($\tau_{Sb-540 \text{ nm}}$) emissions under 254 nm excitation have been resolved by fluorescence decay curves as 2.12 µs and 6.46 µs, as shown on the right in Fig. 4, respectively. These obvious differences in excitation spectra and fluorescence decay curves further confirm the existence of differentiated Sb³⁺ emitting centers in ZASP host glasses.



Fig. 4. Excitation spectra (left) and fluorescence decay curves (right) for 340 nm and 540 nm emissions in ZASP host glasses.



Fig. 5. Emission spectra of Dy³⁺ doped ZASP glasses under excitation of 260 nm. Inserted photos show fluorescence of relevant samples under 254 nm UV lamp radiation.

Emission spectra of Dy^{3+} doped ZASP glasses consist of a broad ~433 nm band of Sb³⁺ and two apparent narrow ~483 nm and ~575 nm bands assigned to ${}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2}$ and ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ transitions of Dy^{3+} , respectively, as shown in Fig. 5. In contrast with the host glasses, the luminescence behavior of Sb³⁺ is still effective in Dy³⁺ doped ZASP glasses. With the increment of Dy³⁺ concentration, the emission intensity of the Sb³⁺ ions decreases clearly while emission intensity of Dy³⁺ ions increases. When the doping concentration of Dy³⁺ exceeds the critical value, the intrinsic luminescence of Dy³⁺ stops growing, as shown in Fig. 5d. With an increase in Dy³⁺ content, the fluorescence color changes from bluish-white to pure white, as presented in the inserted photos of Fig. 5.

3.3. Excitability of high-energy ultraviolet radiation for Dy^{3+}

Excitation spectra for visible emissions of Sb³⁺ and Dy^{3+} are presented in Fig. 6. The group of peaks at 310 nm to 500 nm region for Dv^{3+} emission is assigned to characteristic f-f transition of Dy³⁺. Broad excitation bands in 200 nm to 310 nm region denote that visible emissions from Sb^{3+} and Dy^{3+} can be simultaneously excited and the high consistency of the two series of broad bands reveals the existence of non-radiative energy transfer from Sb^{3+} to Dy^{3+} . The excitation bands of Dy^{3+} are identified to be covering 200 nm to 500 nm region, which is much broader than those from 300 nm to 490 nm in zinc phosphate glasses [58], 375 nm to 550 nm in tellurite glasses [59], 350 nm to 500 nm in bismuth zinc borate glasses [60] and 420 nm to 500 nm in niobium germanate glasses [48]. It is worth noting that the emission band of Sb^{3+} in Fig. 6 possesses a significant overlap with the excitation peaks of Dy^{3+} in the region of 300 nm to 500 nm, suggesting a possible energy transfer from Sb^{3+} to Dy^{3+} . Actually, broad excitation bands in 200 nm to 310 nm region show that visible emissions from Sb^{3+} and Dy^{3+} can be simultaneously excited, and the high consistency of the two series of broad bands reveals the existence of non-radiative energy transfer from Sb³⁺ to Dy^{3+} . In meantime, the excitation spectra extended to UVB and UVC regions further confirm that the Dy³⁺ doped ZASP glasses can be excited by commercially available mercury lamp.



Fig. 6. Excitation spectra of ZASP glasses doped with different concentrations of Dy³⁺ obtained by monitoring 439 nm emission of Sb³⁺ (left) and 575 nm emission of Dy³⁺ (right).

3.4. Absorption spectrum and Judd-Ofelt analysis

The absorption spectrum of Dy³⁺ doped ZASP glasses shows eleven inhomogeneous broadened banks peaking at 364 nm, 386 nm, 425 nm, 453 nm, 473 nm, 752 nm, 801 nm, 898 nm, 1095 nm, 1273 nm and 1680 nm, which are associated with the absorption transitions from the ${}^{6}H_{15/2}$ ground state to the excited states, as labeled in Fig. 7. The radiative transitions belonging to the 4f⁹ electronic configurations can be analyzed by the Judd-Ofelt (J-O) theory based on the absorption of Dy^{3+} [61–69]. J-O intensity parameters Ω_t (t = 2, 4, 6) for Dy^{3+} in ZASP glasses were calculated to be 5.74 $\times 10^{-20}$ cm², 1.47 $\times 10^{-20}$ cm² and 1.12×10^{-20} cm², respectively, and the rootmean-square deviation between the experimental and calculated oscillator strengths was assessed to be 2.3×10^{-7} , indicating that the fitting process is reliable. Ω_2 of Dy³⁺ in ZASP glasses is close to 5.41×10^{-20} cm² in lead fluorogermanate glasses [64], 5.19×10^{-20} cm² in lead tungsten tellurite glasses [65] and 4.90×10^{-20} cm² in lead borate glasses [66], demonstrating a higher asymmetrical and stronger covalent environment around Dy^{3+} in the ZASP glass system.

Using these intensity parameters, the spontaneous emission probability (A_{rad}) and branching ratio (β) were calculated to be 541 s⁻¹ and 64.85 % for the ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ transition, respectively, while they are 101 s⁻¹ and 12.11 % for the



Fig. 7. Absorption spectrum of 8.37 % Dy_2O_3 doped ZASP glasses.

 ${}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2}$ transition. Large A_{rad} and β values are beneficial to the blue and yellow emissions from the ${}^{4}F_{9/2}$ level in Dy³⁺ doped ZASP glasses. The β for ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ transition is 64.9 %, revealing that yellow emission is the dominant radiative component of Dy³⁺.

The experimental lifetimes (τ_{exp}) of the ${}^{4}F_{9/2}$ level of different concentrations Dy^{3+} doped ZASP glasses have been derived from the fluorescence decay curves, as presented in Fig. 8. The quantum efficiencies $\eta_{q} = \tau_{exp}/\tau_{rad}$ for ${}^{4}F_{9/2}$ level have been calculated and listed in Table 1. τ_{exp} can be expressed as:

$$1/\tau_{exp} = 1/\tau_{rad} + W_{MPR} + W_{CR} \tag{3}$$

where W_{MPR} is the multi-phonon relaxation (MPR) rate and W_{CR} is the rate of cross-relaxation (CR) which should be dependent on the ion concentration, and τ_{rad} is the radiative lifetime. The calculated W_{CR} values of different Dy³⁺ concentrations have been derived and summarized in Table 1, where the cross relaxation process at the lowestconcentration is ignored. Although W_{CR} is nonignorable in high concentration doping cases, η_{α} for ${}^{4}F_{9/2}$ level in 3.09 % Dy₂O₃ doping case still reaches 66.7 %, indicating that effective yellow and blue emissions of Dy^{3+} could be expected at reasonable concentration. Meanwhile, the large lifetime and η_{q} enable the Dy³⁺ doped ZASP glasses to be potential candidates for developing visible lasers.



Fig. 8. Fluorescence decay curves for the ${}^{4}F_{9/2}$ level of Dy³⁺ obtained by monitoring 575 nm emissions in 0.26 %, 1.89 %, 3.09 % and 8.37 % Dy₂O₃ doped ZASP glasses under the excitation of 390 nm radiation.

The probability (P) and the efficiency (η) can be derived by:

$$P = 1/\tau - 1/\tau_0$$
 (4)

and:

$$\eta = \frac{P}{1/\tau} = 1 - \frac{\tau}{\tau_0},\tag{5}$$

where τ_0 and τ are the lifetime of the donor in the absence and presence of acceptor, respectively. Fig. 9 shows fluorescence decay curves for the ${}^{3}P_{1}$

level of Sb³⁺ in ZASP glasses doped with different concentrations of Dy³⁺, obtained by monitoring 340 nm and 540 nm emissions. As the concentration of Dy³⁺ increases, the broad fluorescence attributed to Sb^{3+} decreases largely. The P and the η of the non-radiative energy transfer from Sb^{3+} to Dy^{3+} based on monitoring 340 nm and 540 nm emissions are illustrated in Table 2. When monitoring 340 nm emission, P and n values in 3.09 % Dy₂O₃ doping case can be as high as 15.33×10^4 s⁻¹ and 24.53 %, respectively. In contrast, when monitoring 540 nm emission, P and η values are 4.24 × 10⁴ s⁻¹ and 21.52 %, respectively, demonstrating that the radiative energy transfer from Sb^{3+} to Dy^{3+} is efficient. The differences of P and η between the monitoring 340 nm and 540 nm emissions denote that the energy transfer processes between discrepant donor Sb³⁺ and acceptor Dy^{3+} are in-equivalent.

Energy transfer (ET) process from Sb^{3+} to Dy^{3+} in ZASP glasses can be illustrated by energylevel diagram in Fig. 10. Firstly, the UV photons are absorbed by Sb^{3+} ions and the excited energy is stored in the ${}^{3}\text{P}_{1}$ level. Then, the excited Sb^{3+} ions are transferred radiatively to the ground state ${}^{1}\text{S}_{0}$ level, emitting bluish-white light. Meanwhile, a part of energy excited in the ${}^{3}\text{P}_{1}$ of Sb^{3+} could be transferred non-radiatively to Dy^{3+} , after that the excited Dy^{3+} is relaxed non-radiatively to ${}^{4}\text{F}_{9/2}$ level and finally it is transferred radiatively to the ${}^{6}\text{H}_{15/2}$ and ${}^{6}\text{H}_{13/2}$ levels, emitting ~483 nm and ~575 nm lights, respectively. In addition, Dy^{3+} can be pumped directly from the ground state to excited state by absorbing UVA radiation, and then

Table 1. Experimental average lifetime τ_{exp} , quantum efficiencies η_q and cross relaxation rates W_{CR} for different concentrations of Dy^{3+} in ZASP glasses.

Dy ₂ O ₃	τ_{exp}	η_q	W _{MPR}	W _{CR}
[mol%]	[ms]	[%]	$[s^{-1}]$	$[s^{-1}]$
0.26	1.14	95.0	43.9	_
1.89	0.87	72.5	43.9	272.2
3.09	0.80	66.7	43.9	372.8
8.37	0.57	47.5	43.9	877.2



Fig. 9. Fluorescence decay curves for the ³P₁ level of Sb³⁺ obtained by monitoring 340 nm (left) and 540 nm (right) in 1.89 %, 3.09 % and 8.37 % Dy₂O₃ doped ZASP glasses under 254 nm radiation.

Table 2. Fluorescence lifetime τ , energy-transfer probability P and energy-transfer efficiency η from Sb³⁺ in ZASP glasses.

Dy ₂ O ₃ [mol%]	$\tau_{Sb-340} [\mu s]$	$P_{340} [10^4 s^{-1}]$	η ₃₄₀ [%]	$\tau_{Sb-540} [\mu s]$	$P_{540} [10^4 \text{ s}^{-1}]$	η ₅₄₀ [%]
0	2.12	_	_	6.46	_	_
1.89	1.87	6.30	11.79	5.32	3.32	17.65
3.09	1.60	15.33	24.53	5.07	4.24	21.52
8.37	1.12	42.12	47.17	2.92	18.77	54.80



Fig. 10. Energy-level diagram of luminescence centers and possible ET processes from Sb³⁺ to Dy³⁺ under UV radiation.

the excited Dy^{3+} relaxes non-radiatively to the ${}^{4}F_{9/2}$ level and consequently transits to the ${}^{6}H_{15/2}$ and ${}^{6}H_{13/2}$ levels with visible emissions [51].

4. Conclusions

The specificity of the radiation luminescence behavior of Dy^{3+} doped antimony phosphate

(ZASP) glasses is confirmed. The effective excitation region of Dy³⁺ doped ZASP glasses is extended to medium-wave ultraviolet (UVB) and short-wave ultraviolet (UVC) areas. The quantum efficiency for ⁴F_{9/2} level at low- and mediumconcentration Dy₂O₃ doping cases is as high as 95.0 % and 66.7 %, respectively, implying the effectiveness of the emissions from Dy^{3+} in ZASP glasses. The values of energy-transfer probability (P) have significant difference while adopting 340 nm and 540 nm as monitoring wavelengths, so as the energy-transfer efficiencies (η) , which relate to the energy-transfer processes from discrepant Sb^{3+} donors to Dy^{3+} acceptors were in-equivalent. The effective excitability of high-energy ultraviolet radiation demonstrates that Dy³⁺ doped ZASP glasses assisted with Sb^{3+} have a great prospect in developing illumination and display devices.

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