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Upconversion Luminescence of Er^{3+}/Yb^{3+} Co-Doped Sb_2O_3-WO_3-Li_2O Antimonate Glasses

Deqiang Wang, Junjie Lu, Zhen Zhang, Yingfei Hu, Zhiling Shen

Key Laboratory for Ultrafine Materials of Ministry of Education, School of Materials Science and Engineering, East China University of Science and Technology, Shanghai, China.
Email: Derek_wang@ecust.edu.cn

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ABSTRACT

A series of Er^{3+}/Yb^{3+} co-doped Sb_2O_3-WO_3-Li_2O glasses were prepared. Intense green upconversion fluorescence was observed near 524 *and* 544 *nm under excitation at* 980 *nm. The upconversion process was proved to be a two-photon absorption process. The upconversion fluorescence efficiency was enhanced by increasing introduction concentration of Yb^{3+} ions. The low maximum phonon energy of the glasses indicated that the glasses were good potential for upconversion optical devices.*

Keywords*: *Upconversion Fluorescence, Glasses, Laser

1. Introduction

Recently, with the increasing requirement for laser diode (LD), magneto-optical disk and optical media, more and more attention has been focused on searching for luminescence materials with high upconversion efficiency [1-3]. The upconversion is greatly affected by sensitizing combination, pumping laser wavelength and conversion passage, which makes the host materials for rare earth (RE) ions and the dopant ions as the absorption and emission centers very important [4,5]. Glasses have been selected as the potential host materials because of its low phonon energy which can reduce the multiphonon relaxation (MPR) and thus achieves strong upconversion luminescence. Though fluoride glasses have been studied because of their low phonon energies, oxide glasses are more appropriate for practical applications due to their high chemical durability and thermal stability. Although silicate glasses are stable, upconversion fluorescence is difficult to observe in silicate glasses because of its high maximum phonon energy [6]. Nowadays, tellurite and antimonate glasses are of growing interest due to their relative low phonon energy, high refractive index, good corrosion resistance, thermal and chemical stability. The antimonate glasses are more stable against the pumping light, possess high refractive index and are transparent up to the far infrared wavelengths which makes them suitable for hosting the rare earth ions to give out high luminescence efficiency in the visible and NIR regions [7-10].

Triply ionized Er^{3+}, Ho^{3+}, Tm^{3+}, Nd^{3+} ions of the lanthanide series have been widely studied for upconversion processes in various glass hosts. Erbium ion (Er^{3+}) has been recognized as one of the most efficient ions for obtaining frequency upconversion [11,12]. In order to improve the pumping efficiency of 980 nm LD, the sensitization of Er^{3+} doped materials with Yb^{3+} ions is a popular way to increase the optical pumping efficiency because Yb^{3+} ions exhibits an intense broad absorption cross section between 870 and 1050 nm [13], while Er^{3+} has low absorption at 980 nm.

In this paper, we report our progress on the fabrication and upconversion luminescence characterization of a novel Er^{3+}/Yb^{3+} co-doped Sb_2O_3-WO_3-Li_2O (SWL) glasses. The phonon energy of this glass has been predicted from the FT-IR spectra. The purpose of this paper is to develop a new antimony glass with low phonon energy, and understanding of the upconversion behavior in this glassy host for predicting its potential laser properties.

2. Experimental

Antimonate glasses were prepared by melting the reagent grade Sb_2O_3, Li_2CO_3, WO_3, Er_2O_3 and Yb_2O_3 as the starting materials. The starting materials were sufficiently mixed and grinded, then were melted at 1000˚C -

1100˚C for 10 min, followed by an annealing at 270˚C for 3 h in a muffle furnace to eliminate the internal stress, then slowly cooled down to the room temperature. Compositions chosen in the present study are shown in **Table 1**. Finally, glass samples were cut, ground and polished for the following measurement.

Thermal stability analyses of the glasses were determined by using a CRY-2CRY-1WRT-1 thermal analysis (DTA) at a heating rate of 10˚C /min from room temperature to 800˚C. IR transmission spectra were recorded between 400 and 4000 cm^{-1} (Nicolet 6700). UV transmission spectra were recorded between 300 and 1000 nm (Shanghai Lengguang S54). For Er^{3+}/Yb^{3+} co-doped antimonate bulk glasses, the upconversion luminescence spectra were obtained with a spectrofluorimeter (Jobin Yvon Fluorolog3-p, France) upon excitation of 980 nm LD with a maximum power of 1 W. The glass was positioned so that the pump beam was allowed to be incident at the edge of the glass sample, and the optical path of emitted light through the sample to the detector was approximately 1 mm.

3. Results and Discussion

3.1. DTA and XRD Spectra

The DTA spectra of $0.25Er^{3+}/0.75Yb^{3+}$ co-doped Sb_2O_3-WO_3-Li_2O glasses (mol%) (No. 7) was shown in **Figure 1**. From the figure, it can be seen that the transition temperature and melting temperature of this glass is 272˚C and 584˚C respectively. The 550˚C point is the beginning of melting temperature point. There is an obvious crystallization peak at 385˚C (Tx). The difference between the glass transition temperature (Tg) and the onset crystallization temperature (Tx), $\Delta T = Tx - Tg$ has been frequently quoted as a rough indicator of glass stability against crystallization [14-16]. It is desirable for a glass host to have a ΔT as large as possible. ΔT here is 113˚C ($\Delta T = Tx - Tg = 385 - 272 = 113$˚C), indicating that the SWL glasses have fairly good thermal stability and are capable for further performing fabrication and crystal- ree fiber drawing.

Table 1. The composition of Er^{3+} doped Sb_2O_3-Li_2O-WO_3 glasses and Er^{3+}/Yb^{3+} codoped Sb_2O_3-Li_2O-WO_3 glasses (mol%).

No.	Sb_2O_3	WO_3	LiO	Er_2O_3	Yb_2O_3
1	80	10	10	0.25	-
2	80	10	10	0.50	-
3	80	10	10	0.75	-
4	80	10	10	1.00	-
5	80	10	10	0.25	0.25
6	80	10	10	0.25	0.50
7	80	10	10	0.25	0.75
8	80	10	10	0.25	1.00

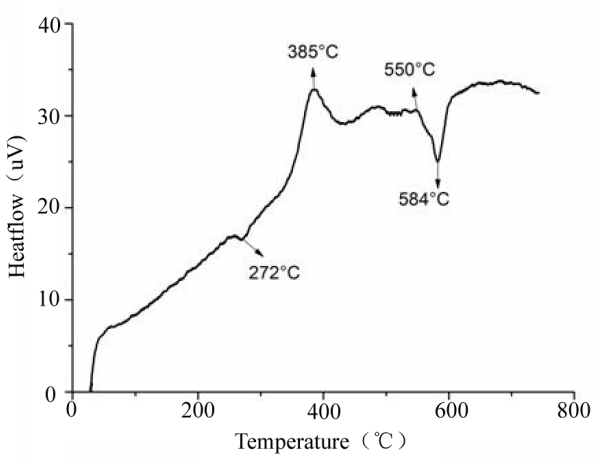

Figure 1. The DTA spectra of $0.25Er^{3+}/0.75Yb^{3+}$ codoped Sb_2O_3-Li_2O-WO_3 glasses (mol%).

3.2. Absorption Spectra

Figure 2 showed IR spectra of $0.25Er^{3+}/0.25Yb^{3+}$ and $0.25Er^{3+}/0.75Yb^{3+}$ co-doped SWL glasses. The absorption band near 947 and 697 cm^{-1} is attributed to the vibration of W-O and W-O-W respectively. The absorption band near 600 cm^{-1} is attributed to symmetric bending vibrations of Sb–O–Sb and the absorption band near 480 cm^{-1} is attributed to doubly degenerate bending vibrations of [SbO_3] structural units. In the glasses, Sb^{3+} ions form a threefold coordination environment with oxygen and Sb^{3+} behaves as a classic network-forming cation in oxide glasses, creating a continuous random network of Sb–O–Sb. The position of the highest phonon band is important because the multi-phonon decay of rare-earth ions in a glass depends on the maximum phonon energy of the host glass [17,18]. In this kind of antimonate glasses, the highest band (600 cm^{-1}) could be attributed to the vibration of W-O. The maximum phonon energy of the glass is low [10,19]. Therefore, it can be expected that Sb_2O_3-WO_3-Li_2O glasses are good candidates for fabrication of upconversion optical devices.

The absorption spectra of the Er^{3+}/Yb^{3+} co-doped antimonate bulk glass in the visible region was shown in **Figure 3**. Four absorption bands are shown by the excited levels, which are attributed to the transitions from the ground state ($^4I_{15/2}$) to the excited state of Er^{3+} ions: $^4F_{7/2}$, $^2H_{11/2}$, $^4F_{9/2}$ and $^4I_{11/2}$ respectively [20]. The absorption at the wavelength region at 980 nm is due to the large contribution of the absorption of Yb^{3+}, which arises from the $^2F_{7/2} \rightarrow {}^2F_{5/2}$ transition. Upon the introduction of Yb^{3+} ions to Er^{3+} doped antimonate glasses, the absorption efficiency at about 980 nm bands is enhanced by energy transfer process Er^{3+}: $4I_{15/2}$ + Yb^{3+}: $^2F_{5/2}$ → Er^{3+}:

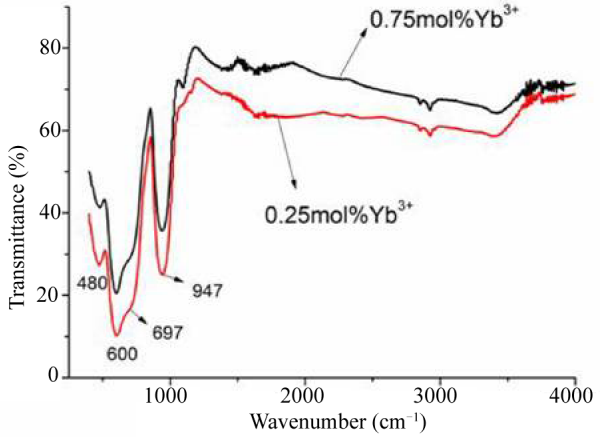

Figure 2. IR spectra of the 0.25Er^{3+}/Yb^{3+} codoped Sb_2O_3-Li_2O-WO_3 glasses (mol%).

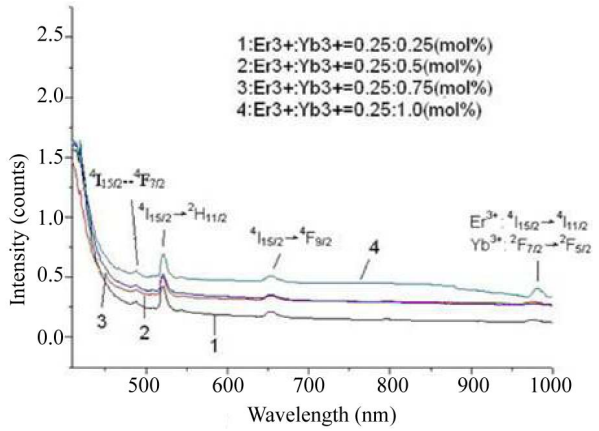

Figure 3. The adsorption spectra of Er^{3+}/Yb^{3+} codoped Sb_2O_3-Li_2O-WO_3 glasses.

$^4I_{11/2}$ +Yb^{3+}: $^2F_{7/2}$. The results at this condition will make more Er^{3+} ions involving the pumped process [21,22].

3.3. Upconversion Fluorescence Spectra

Figure 4 illustrated the upconversion emission spectra of the 0.25 mol% Er^{3+}, 0.5 mol% Er^{3+}, 0.75 mol% Er^{3+} and 1.00 mol% Er^{3+} single doped SWL glasses in the wavelength range of 500 - 700 nm with 980 nm LD under the same powder 1062.6 mW excitation. The observed upconversion luminescence in the green spectral bands has three humped peaks at 524, 544 and 656 nm wavelength are attributed to the Er^{3+}: $^2H_{11/2} \rightarrow {}^4I_{15/2}$, $^{4S3}/_2 \rightarrow {}^4I_{15/2}$ and $^4F_{9/2} \rightarrow {}^4I_{15/2}$ transitions respectively. The intensity gain increases with the increasing of Er^{3+} concentration from 0.25mol% to 0.75mol%, while decrease significantly when the Er^{3+} concentration reaches 1.00 mol% due to upconversion fluorescence quenching of Er^{3+} ions.

The upconversion emission spectra of 0.75 mol% Er^{3+}

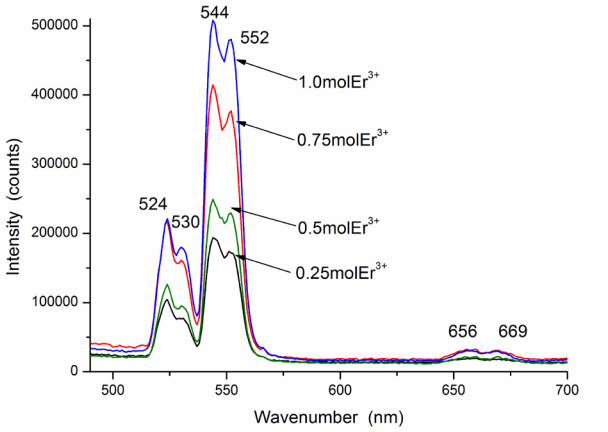

Figure 4. The upconversion emission spectra of the (a) 0.25 mol% Er^{3+}, (b) 0.5 mol% Er^{3+}, (c) 0.75 mol% Er^{3+} and (d) 1.00 mol% Er^{3+} doped Sb_2O_3-Li_2O-WO_3 glasses in the wavelength range of 500 - 700 nm with 980 nm LD under the same powder 1062.6 mW (CI: 20 mA).

doped SWL glasses in the wavelength range of 500 - 700 nm with 980 nm LD under different powder are shown in **Figure 5**. The LD current intensity (CI) is varied from 10 mA to 20 mA every 2 mA, the corresponding power is 372.6 mW, 510.6 mW, 648.6 mW, 786.6 mW, 924.6 mW and 1062.6 mW.

Figure 6 shows the log-log dependences of the integrated green (524 and 544 nm) intensities on the excitation power at 980 nm. In frequency upconversion process, the upconversion emission intensity Iup increases in proportion to the input power of infrared (IR) excitation intensity IIR, that is, Iup $\propto$ IIR n, where n is the number of IR photons absorbed per visible photon emitted. A plot of log Iup vs. log IIR yields a straight line with slope n. The quadratic dependence indicates that two photons are involved for the upconversion process.

The upconversion emission spectra of Sb_2O_3-WO_3-Li_2O bulk glasses and the 0.25 mol% Er^{3+}/0.25 mol% Yb^{3+}, 0.25 mol% Er^{3+}/0.5 mol% Yb^{3+}, 0.25 mol% Er^{3+} /0.75 mol% Yb^{3+} and 0.25 mol% Er^{3+}/1.00 mol% Yb^{3+} co-doped Sb_2O_3-Li_2O-WO_3 glasses in the wavelength range of 500 - 700 nm with 980 nm LD under the same powder 1062.6 mW were shown in **Figure 7**. **Figure 8** showed the log dependences of the integrated green (524 and 544 nm) intensities on the amount of Yb^{3+} ions. From the **Figure 8**, it can be seen that the addition of small amount of Yb^{3+} ions to the glass containing Er^{3+} enhances the emission intensity of Er^{3+} ions 3 - 5 times for 524 and 544 nm bands respectively and subsequently the fluorescence intensity of Er^{3+} ions.

3.4. Upconversion Mechanisms Analysis

Based on the **Figures 4-8** and according to the precious

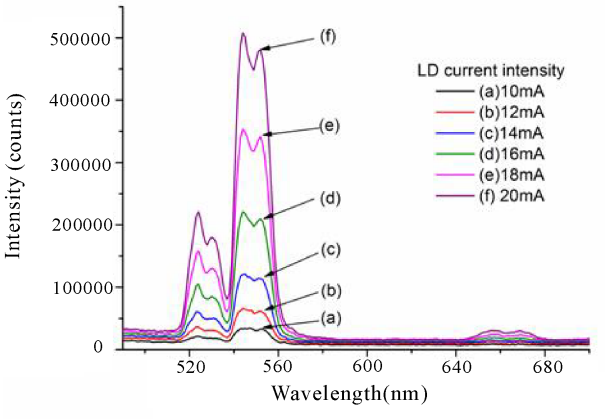

Figure 5. The upconversion emission spectra of 0.75 mol% Er^{3+} doped Sb_2O_3-Li_2O-WO_3 glasses in the wavelength range of 500 - 700 nm with 980 nm LD.

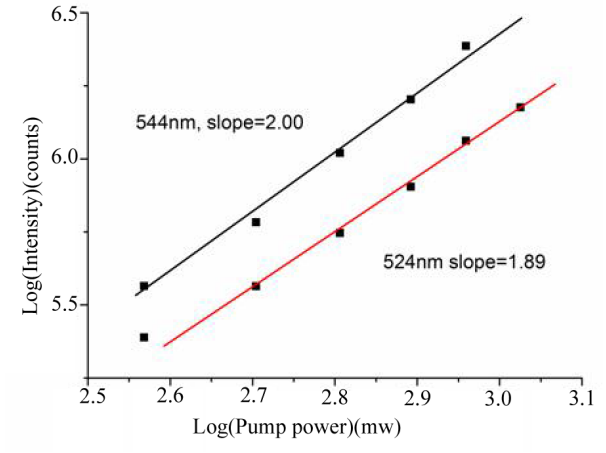

Figure 6. The log–log dependences of the integrated green (524 and 544 nm) intensities on the excitation power at 980 nm.

reports, the mechanisms of energy transfer from Yb^{3+} to Er^{3+} can be described as **Figure 9**. For the green emission, in the first step, the $^4I_{15/2}$ level is directly excited with 980 nm light as follows:

$$^4F_{15/2}\left(Er^{3+}\right)+a.hpoton \rightarrow {}^4H_{11/2}\left(Er^{3+}\right) \quad (1)$$

$$^2F_{7/2}\left(Yb^{3+}\right)+a.hpoton \rightarrow {}^2F_{5/2}\left(Yb^{3+}\right) \quad (2)$$

An incident 980 nm photon is strongly absorbed by Yb^{3+} ions and excites them to $^2F_{5/2}$ level along with the direct absorption of Er^{3+} ions. The excited Yb^{3+} ions transfer their excitation energy to unexcited Er^{3+} ions, promoting them to $^4I_{11/2}$ level thus enhancing the poplation of $^4I_{11/2}$ level further [23,24]. Thus, the second step involves as follows:

$$^4I_{11/2}\left(Er^{3+}\right)+a.hpoton \rightarrow {}^4F_{7/2}\left(Er^{3+}\right) \quad (3)$$

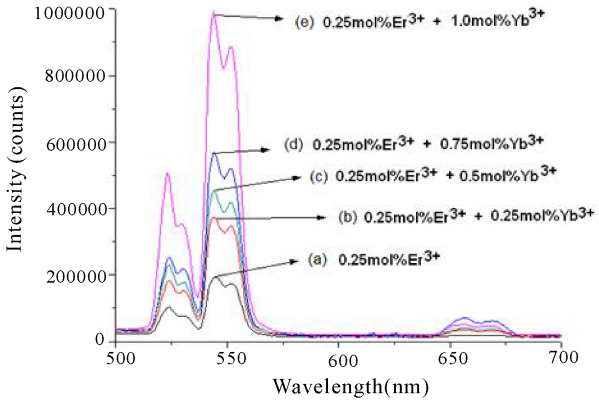

Figure 7. The upconversion emission spectra of (a) 0.25 mol% Er^{3+} and (b) 0.25 mol% Er^{3+}/0.25 mol% Yb^{3+}, (c) 0.25 mol% Er^{3+}/0.5 mol% Yb^{3+}, (d) 0.25 mol% Er^{3+}/0.75 mol% Yb^{3+} and (e) 0.25 mol% Er^{3+}/1.00 mol% Yb^{3+} codoped Sb_2O_3-Li_2O-WO_3 glasses in the wavelength range of 500 - 700 nm with 980 nm LD under the same powder 1062.6 mW (CI: 20 mA).

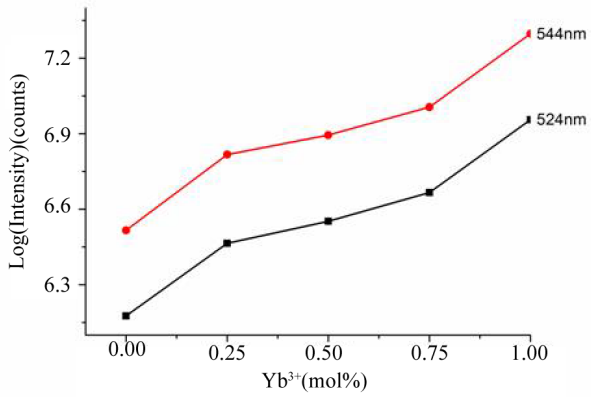

Figure 8. The dependences of the integrated green (524 and 544 nm) intensities on the amount of Yb^{3+} ions.

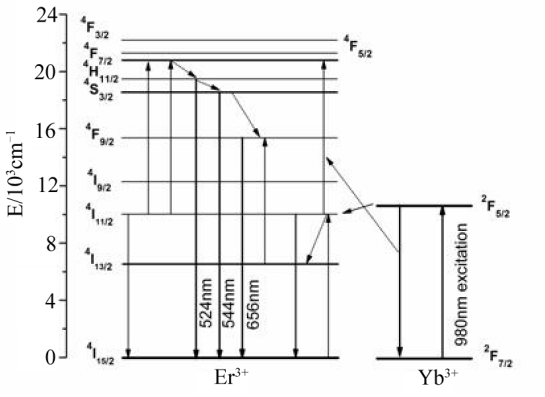

Figure 9. Energy level diagram of Er^{3+}/Yb^{3+} and upconversion mechanisms of Sb_2O_3-Li_2O-WO_3 glasses under 980 nm excitation power.

$$^2F_{5/2}\left(Yb^{3+}\right)+{}^4I_{11/2}\left(Er^{3+}\right)\rightarrow {}^2F_{7/2}\left(Yb^{3+}\right)+{}^4F_{7/2}\left(Er^{3+}\right) \quad (4)$$

The populated $^4F_{7/2}$ level of Er^{3+} then relaxes rapidly and non-radiatively to the next lower levels, $^2H_{11/2}$ and $^4S_{3/2}$. The above processes then produce the two transitions $^2H_{11/2}$-$^4I_{15/2}$ and $^4S_{3/2}$-$^4I_{15/2}$, which are centered at 524 and 544 nm respectively. The trace presence of Yb^{3+} ions provides an additional channel to populate Er^{3+} ion levels, make more Er^{3+} ions involving the pumped process and thus enhance the green fluoroscence intensity.

For the red emission, it can be seen from **Figures 4**, **5** and **7**, the upconversion luminescence in the red spectral bands at 650 - 670 nm wavelength is very weak, which means few Er^{3+} ions is involving the following processes:

$$^4I_{11/2}\left(Er^{3+}\right)+a.hpoton\rightarrow {}^4F_{7/2}\left(Er^{3+}\right) \quad (5)$$

$$^4I_{11/2}\left(Er^{3+}\right)\rightarrow {}^4I_{13/2}\left(Er^{3+}\right)+a.hpoton \quad (6)$$

$$^4I_{13/2}\left(Er^{3+}\right)+a.hpoton\rightarrow {}^4F_{9/2}\left(Er^{3+}\right) \quad (7)$$

4. Conclusions

A series of Er^{3+}/Yb^{3+} co-doped Sb_2O_3-WO_3-Li_2O glasses were prepared. Intense green upconversion fluorescence was observed near 524 and 544 nm in the Er^{3+}/Yb^{3+} co-doped Sb_2O_3-WO_3-Li_2O glasses under 980 nm excitation. The upconversion processes were proved to involve the sequential two-photon absorption process for the green emissions. The maximum phonon energy of the glass is about 348 cm^{-1}, which is much lower than silicate glasses. Upon the introduction of Yb^{3+} ions to Er^{3+} doped antimonate glasses, the upconversion fluorescence efficiency is enhanced by increasing of Yb^{3+} concentration. The upconversion excitation increased with the increasing of LD powder. The data presented in this work might provide useful information for further development of Er^{3+}-doped materials for upconversion optical devices.

5. Acknowledgements

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