

# Luminescent properties of (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> under VUV excitation for PDP prepared by co-precipitation method

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The (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors were prepared by co-precipitation method combined with ultrasonic-disperse with Y(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O, Gd(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O, Eu(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O and H<sub>3</sub>BO<sub>3</sub> as the starting material, oxalic acid as precipitant. The samples were characterized by thermo-gravimetric analyzer, X-ray powder diffraction and Vacuum Ultraviolet (VUV) test system for plasma display panel. The (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors are found to have the best luminous performance at 611 nm with respect to intensity and color purity under VUV excitation. At the same time, the (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors prepared by co-precipitation method show high crystallinity, high distribution and good luminescence intensity.

(Received March 21, 2011; accepted April 11, 2011)

**Keywords:** Co-precipitation method, (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors, Excitation spectra, Emission spectra

## 1. Introduction

Along with the development of technology and improving the quality of people's life, people are looking forward to developing high-definition flat panel display screen. Compared with the other display models such as the cathode-ray tube, vacuum fluorescent display, field emission display, liquid crystal display and electroluminescent display, plasma display panel (PDP) has shown considerable potential advantages when they acted as flat panel display screen, such as high definition, good chemical stability, wide visual angle, large screen, low weight, high intensity irradiation and good color purity, etc [1, 2]. Because those advantages, the technology of PDP has been edged competitively in the market of large-sized flat panel display by the end of last century.

The emission mechanism of PDP is that the vacuum ultraviolet light (VUV light) produce by the gas discharge at the display screen, then the VUV light stimulates the phosphors of three primary colors (red, green, blue), and the whole color display is achieved [3]. So PDP phosphors are one of the important factors influencing the PDP device performance. The excitation spectra, emission spectra, luminescent color, persistence time, optical attenuation and quantum efficiency of photoluminescence are the important marks of PDP phosphors. In order to have a good picture quality, much attentions has been paid to improve and develop PDP phosphors. According to the different composition, the PDP phosphors concluded five styles, which were silicate category phosphors [4] aluminates category phosphors [5, 6], borate category phosphors [7], phosphate category phosphors [8, 9] and other compound phosphors [10-12].

Because of the special of red light, the red PDP

phosphors have still problems with respect to the preparation and performance for wide application. At present, the PDP phosphors mainly contained (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> [13], Y<sub>2</sub>O<sub>3</sub>:Eu<sup>3+</sup> [14] and YBO<sub>3</sub>:Eu<sup>3+</sup> [15] and so on. Because the (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> red phosphors have the maximum luminescent, it is the best choice among those red phosphors when excited by the VUV light. Conventionally, phosphor materials are obtained by solid-state reaction (SSR) at high temperature. However, during the solid-state reaction process, it is difficult to control over particles size and morphology so that the phosphor suffers for a long time calcination at very high temperature with the result that its particles have large size (2-5 μm) with wide distribution range and heterogeneous morphology, which results in unfavorable luminescent properties in the applied devices [16]. In order to improve the characteristics of the phosphor, much attention have been paid to the synthesis of red phosphor and various synthesis techniques have been developed to prepare high-quality YGB red phosphors [17], such as combustion synthesis method [18], co-precipitation [19-21], hydrothermal method [22], spray-pyrolysis method [23], sol-gel method [24], and so on. All these methods can be classified into soft chemistry method due to its reaction in a solution. In these methods, the co-precipitation method has been considered as a common method for preparing YGB red phosphor, due to its many advantages, such as low cost, low reaction temperature, simple equipment and less consumption of energy. Meanwhile, the YGB red phosphors prepared by the co-precipitation method have better luminous performance than other methods.

In this paper, (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors were prepared by co-precipitation reaction. The thermal decomposition of the precursor and the crystalline phase of the (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors were investigated by

Thermogravimetry/Differential Thermal Analysis (TG/DTA) and X-ray powder diffraction (XRD), respectively. Dependence of luminous properties on concentration of Europium and Gadolinium under VUV excitation was discussed by means of excitation and emission spectroscopy.

## 2. Experimental

### 2.1 Chemicals

Yttrium nitrate (Y(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O) (Tianjin Fuchen Chemical Reagents Co.), gadolinium nitrate (Gd(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O) (Tianjin Kemiou Chemical Reagents Co.), europium nitrate (Eu(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O) (China Pharmaceutical Group Shanghai Chemical Reagent Co.), oxalic acid (H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>) (Tianjin Regent Chemicals Co.), boric acid (H<sub>3</sub>BO<sub>3</sub>) (Shanghai Chemical Reagents Co.), ammonia (NH<sub>3</sub>•H<sub>2</sub>O) (Qingdao Chemical Reagents Co.), ethanol (CH<sub>3</sub>CH<sub>2</sub>OH) (Laiyang Economic and Technological Development Zone Fine Chemical Co.) used in the present experiments were all analytically pure grade.

### 2.2 Preparation of (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup>

Precipitation processes were performed at room temperature. The reactants consisting of Y(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O, Gd(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O, Eu(NO<sub>3</sub>)<sub>3</sub>•6H<sub>2</sub>O in different stoichiometric ratio were completely dissolved in de-ionized water by stirred, then the solution was stayed under ultrasound for 20 min (Solution A). 2.0714g H<sub>3</sub>BO<sub>3</sub> and 5.0075g H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> were dissolved in de-ionized water completely (Solution B). Solutions A and B were dropped in the same reactor and agitated vigorously. In this process, the pH was adjusted between 8 and 9 by NH<sub>3</sub>•H<sub>2</sub>O. After cooled to room temperature, the turbid aspiration was vibrated by ultrasound for 1h, and then filtered and washed 4 or 5 times by de-ionized water and ethanol, respectively. After that, the obtained samples were dried at 100 °C for 12 h to get white powders. Finally, the white powders were calcined at 600 °C for 1h, and 950 °C for 2h. After cooled to room temperature naturally, the white powders of (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors were obtained. Table 1 shows the different rare earth doped concentrations in the preparation of (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors.

Table 1. The samples of different rare earth doped concentration.

number	Molecular formula	Y(NO <sub>3</sub> ) <sub>3</sub> •6H <sub>2</sub> O m/g	Gd(NO <sub>3</sub> ) <sub>3</sub> •6H <sub>2</sub> O	Eu(NO <sub>3</sub> ) <sub>3</sub> •6H <sub>2</sub> O	H <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	H <sub>3</sub> BO <sub>3</sub>
1	(Y <sub>0.85</sub> Gd <sub>0.1</sub> )BO <sub>3</sub> :Eu <sub>0.05</sub>	13.0224	1.8051	0.8920	5.0065	2.4730
2	(Y <sub>0.83</sub> Gd <sub>0.1</sub> )BO <sub>3</sub> :Eu <sub>0.07</sub>	12.7149	1.8048	1.2487	5.0074	2.4731
3	(Y <sub>0.8</sub> Gd <sub>0.1</sub> )BO <sub>3</sub> :Eu <sub>0.1</sub>	12.2552	1.8056	1.7836	5.0079	2.4733
4	(Y <sub>0.7</sub> Gd <sub>0.1</sub> )BO <sub>3</sub> :Eu <sub>0.2</sub>	10.7261	1.8049	3.5679	5.0078	2.4732
5	(Y <sub>0.9</sub> Gd <sub>0.05</sub> )BO <sub>3</sub> :Eu <sub>0.05</sub>	13.7874	0.9027	0.8925	5.0076	2.4734
6	(Y <sub>0.8</sub> Gd <sub>0.15</sub> )BO <sub>3</sub> :Eu <sub>0.05</sub>	12.2553	2.7081	0.8929	5.0070	2.4731
7	(Y <sub>0.75</sub> Gd <sub>0.2</sub> )BO <sub>3</sub> :Eu <sub>0.05</sub>	11.4902	3.6179	0.8927	5.0073	2.4730

### 2.3 Characterization of samples

The thermal decomposition of the (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors precursor was investigated by thermo-gravimetric analyzer (STA409PC, NETZSCH, Germany) using Al<sub>2</sub>O<sub>3</sub> as reference. The heating rate is 20 °C/min under nitrogen atmosphere. The crystalline phase of the (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors was analyzed by Rigaku D/Max-2500 VPC diffractometer with CuKα radiation (λ=1.54056Å) operated at 40 kV and 200 mA in the rang of 10-80° (2θ). The excitation and emission spectra of (Y, Gd)BO<sub>3</sub>:Eu<sup>3+</sup> phosphors under the condition of VUV light were evaluated by beamline 4B8 at the Beijing Synchrotron Radiation Facility (BSRF). The excitation wavelength was 214nm and the wavelengths of emission and excitation spectra were collected from 550 to 750nm and from 125 nm to 350 nm, respectively.

## 3. Result and discussion

### 3.1 TG-DTA curve analysis

The thermal decomposition and weight change behavior were studied with differential thermal gravimetric analysis at a heating rate of 20 °C/min from 20 °C to 900 °C. The result was shown in Fig. 1.

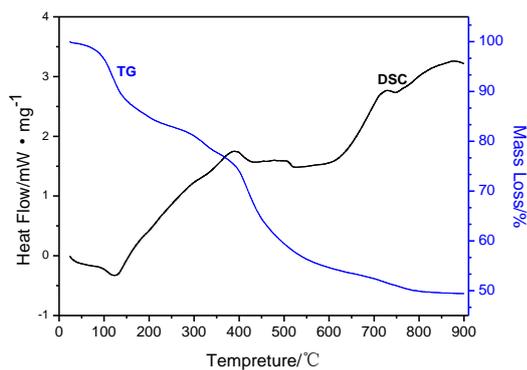


Fig. 1. TG-DTA curves of  $(Y, Gd)BO_3:Eu^{3+}$  phosphors precursors.

There is a process of losing water in the range of 20 °C-160 °C. The weight loss of precursors is about 12.15% on the Thermogravimetry (TG) curve, the corresponding Differential Scanning Calorimetry (DSC) curve at 125 °C has a decalescence peak, which is mainly due to the evaporation of surface absorbed water and the vestigial ethanol. The second weight loss of precursors is likely 14.1%, which is due to the combustion of  $H_2C_2O_4$ . From the corresponding DSC curve, it can be seen that the whole process is an exothermic process from 160 °C to 400 °C. Between 400 °C and 600 °C, the third weight loss of precursors is the decomposition of  $Y(NO_3)_3 \cdot 6H_2O$ ,  $Gd(NO_3)_3 \cdot 6H_2O$ ,  $Eu(NO_3)_3 \cdot 6H_2O$  and the combustion of residue of  $H_2C_2O_4$  and  $H_3BO_3$ . Because of combustion of organic matter, the corresponding DSC curve shows that this process is exothermic process. From 600 °C to 800 °C it can be seen that changing of weight is not obvious. The reason is that the decomposition of  $Y(NO_3)_3 \cdot 6H_2O$ ,  $Gd(NO_3)_3 \cdot 6H_2O$ ,  $Eu(NO_3)_3 \cdot 6H_2O$  and the combustion of residual organic matter has been finished, and the sample has begun to crystallize. Therefore, referencing relative papers [20, 21] and the TG-DTA curves, the presintering temperature was selected at 600 °C for combustion of organic matter and the calcined temperature was selected at 950 °C for crystallizing of  $(Y, Gd)BO_3:Eu^{3+}$  phosphors in our experimental.

### 3.2 XRD analysis

The  $(Y, Gd)BO_3:Eu^{3+}$  phosphors prepared by co-precipitation method were analyzed by XRD, and their XRD patterns are shown in Fig. 2. From this figure, it can be seen that diffraction peaks are very obvious. Compare with the standard patterns (JCPDS card No.13-0531), the XRD pattern of prepared  $(Y, Gd)BO_3:Eu^{3+}$  phosphors are consistent with the standard patterns mostly. The diffraction peaks are narrow and diffraction intensity is high, which shows that the prepared  $(Y, Gd)BO_3:Eu^{3+}$  phosphors are perfectly crystallized. However, some diffraction peaks have a little different intensity and location showing that some impurity exists in the lattice. The reason is probably that the  $Gd^{3+}$  ion is entered into

$YBO_3$  lattice, which leads to the enlargement of crystal lattice. In conclusion, the prepared  $(Y, Gd)BO_3:Eu^{3+}$  phosphors have the perfect crystallization and belong to the hexagonal system.

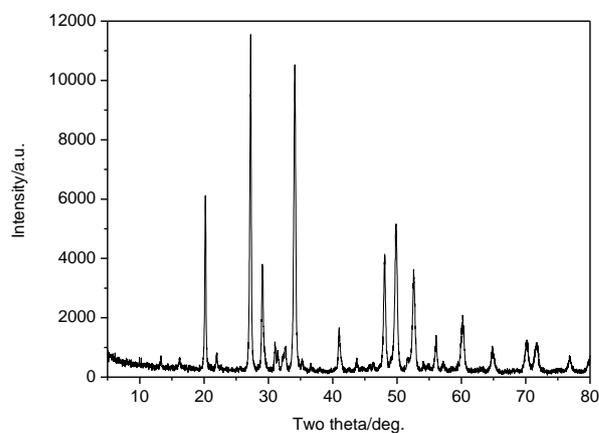


Fig. 2. XRD patterns of  $(Y, Gd)BO_3:Eu^{3+}$  phosphors.

### 3.3 Luminescence property of $(Y, Gd)BO_3:Eu^{3+}$ phosphors

#### 3.3.1 Excitation spectra of $(Y, Gd)BO_3:Eu^{3+}$ phosphors under VUV

Fig. 3 shows the excitation spectra of different Eu-doped  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors prepared by the co-precipitation method. From Fig. 3, it can be seen that the location and shape of the excitation spectra of  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors change with the change of Eu doping concentration. With the increase of concentration of Eu, the relative intensity decreases and the excitation peak of 214 nm is disappeared. When the concentration of Eu-doped is relatively low ( $x=0.05$ ), the intensity of excitation spectra shows the maximum value.

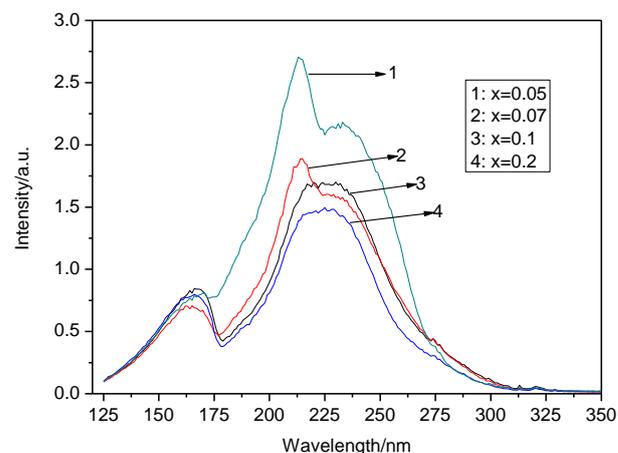


Fig. 3. The excitation spectra of Eu-doped  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors.

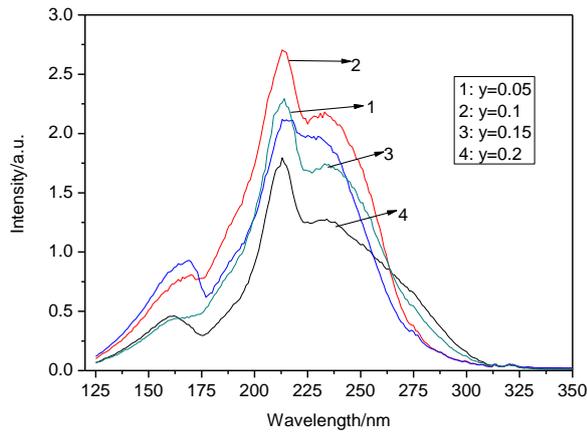


Fig. 4. The excitation spectra of Gd-doped  $(Y_{0.95-y}Gd_y)BO_3:Eu_{0.05}$  phosphors.

Fig. 4 shows the excitation spectra of different Gd-doped  $(Y_{0.95-y}Gd_y)BO_3:Eu_{0.05}$  phosphors prepared by the co-precipitation method. From Fig. 4, it can be seen that the location and shape of the excitation spectra of  $(Y_{0.95-y}Gd_y)BO_3:Eu_{0.05}$  phosphors unchange with the change of Eu doping concentration. However, the relative intensity increases with an increase of Gd concentration when the concentration of Gd-doped is relatively low ( $y < 0.1$ ). By contrast, when  $y > 0.1$ , the relative intensity decreases and the maximum value appears in  $y = 0.1$ , which shows the best excitation properties.

As is shown in the Fig. 4, the excitation spectra have different excitation intensities with different concentration of gadolinium under VUV excitation. There are two wide excitation bands at 165 nm and 214 nm, which can be attributed to the absorption due to charge transfer transition from the  $2p^6$  orbit of ligand  $O^{2-}$  to the  $4f^6$  of  $Eu^{3+}$  empty orbit.

### 3.3.2 Emission spectra of $(Y, Gd)BO_3:Eu^{3+}$ phosphors under VUV

Fig. 5 shows emission spectra of different Eu-doped  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors prepared by the co-precipitation method. For  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors, there are three emission peaks at 593 nm, 611 nm and 627 nm, respectively. The intensity of emission peak of 593 nm exhibits the biggest change in comparison with those of other two peaks with a decrease of the Eu doping concentration. However, the intensity of three emission peaks decreases with the increase of the Eu doping concentration.

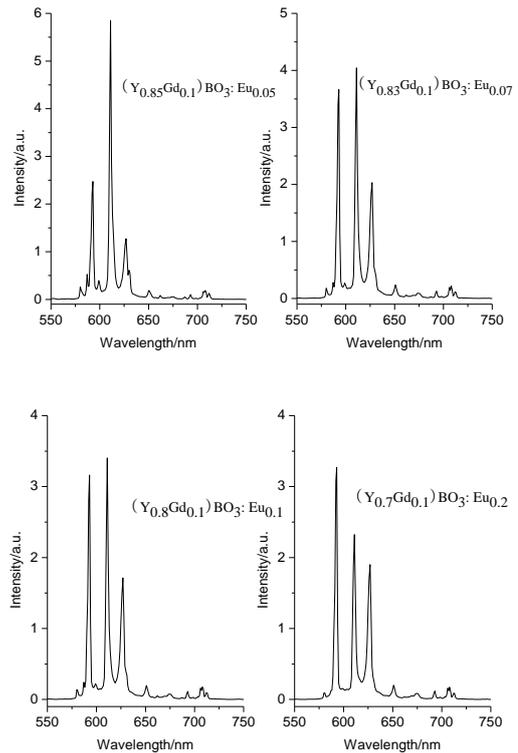


Fig. 5. The emission spectra of different Eu-doped  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors.

The Eu doping concentration is the most important factor for luminescent properties of  $(Y, Gd)BO_3:Eu^{3+}$  phosphors. The three sharp emissions peaked at 593 nm, 611 nm and 627 nm originate from transitions of  ${}^5D_0 \rightarrow {}^7F_1$ ,  ${}^5D_0 \rightarrow {}^7F_2$  and  ${}^5D_0 \rightarrow {}^7F_3$ , respectively. When the doping concentration of Eu is 0.05, the emission peak at 611 nm ( ${}^5D_0 \rightarrow {}^7F_2$ ) is the main emission peak. When increasing Eu, intensity of peak at 593 nm ( ${}^5D_0 \rightarrow {}^7F_1$ ) increase and becomes dominant at the Eu concentration of 0.2. The peak at 593 nm shows the orange color. Because the orange color influences the excitation purity of red emission, the application of  $(Y, Gd)BO_3:Eu^{3+}$  phosphors is limited in excitation of VUV light. So when the doping concentration of Eu is 0.05, the optimum excitation purity of red emission is obtained.

Fig. 6 shows the emission spectra of different Gd-doped  $(Y_{0.95-y}Gd_y)BO_3:Eu_{0.05}$  phosphors prepared by the co-precipitation method. As is shown in Fig. 6, there are three emission peaks for  $(Y_{0.9-x}Gd_{0.1})BO_3:Eu_x$  phosphors too, which belong to 593 nm, 611 nm and 627 nm, respectively. Among four emission spectra, the emission peak at 611 nm is always the highest emission peak. However, the intensity of three emission peaks reduces with the increase of Gd doping concentration.

Three sharp emission peaks at 593 nm, 611 nm and 627 nm are due to the transitions of  ${}^5D_0 \rightarrow {}^7F_1$ ,  ${}^5D_0 \rightarrow {}^7F_2$ , and  ${}^5D_0 \rightarrow {}^7F_3$ , respectively. When the Gd doping concentration is 0.1, the emission peak at 611 nm ( ${}^5D_0 \rightarrow {}^7F_2$ ) is the main emission peak. With increasing Gd doping concentration, the intensity of emission peak at 593 nm increases relatively. This phenomenon has

disadvantages for the excitation purity of red emission. Therefore, the  $(Y_{0.9}Gd_{0.05})BO_3:Eu_{0.05}$  phosphor has better color purity assumed as a red PDP phosphors candidate, though its intensity is lower than the  $(Y_{0.85}Gd_{0.1})BO_3:Eu_{0.05}$  phosphors.

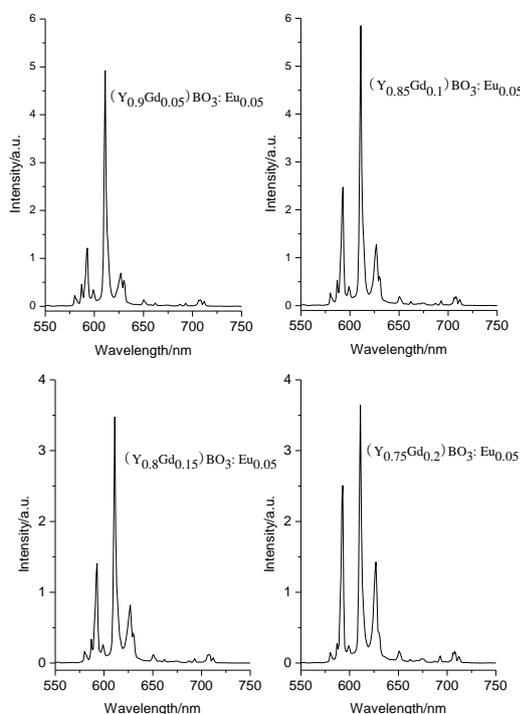


Fig. 6. The emission spectra of Gd-doped  $(Y_{0.95-3}Gd_{3})BO_3:Eu_{0.05}$  phosphors.

#### 4. Conclusions

$(Y, Gd)BO_3:Eu^{3+}$  phosphors have been prepared by the co-precipitation method combined with ultrasonic-disperse and are characterized by TG-DSC, XRD and VUV test system for PDP. The phosphor prepared by this method shows many desirable characteristics such as high crystallinity, high distribution, and good photoluminescence intensity. The luminescence of red phosphor  $(Y, Gd)BO_3:Eu^{3+}$  is influenced by the mole ratio of Y, Gd and Eu. With respect to luminescence intensity and color purity under VUV excitation,  $(Y_{0.9}Gd_{0.05})BO_3:Eu_{0.05}$  phosphor shows the best luminous performance at 611 nm.

#### Acknowledgements

We acknowledge financial support from China Postdoctoral Science Foundation (20100471663), Science and Technology Program of Yantai City (2008151), Natural Science Foundation of Shandong Province (No. ZR2009BL013) and Innovation Group Foundation Plan of Ludong University.

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