

CHAPTER V

CONCLUSIONS

This work presents the preparation of ZrO₂ crystals doped with Eu and Y by using wet chemical method. The mole ratios of Y and Eu were varied while the mole ratio of Zr was kept constant. The calcinations temperature was varied from 600 °C to 1000 °C. Different types of chelating agents were used. Our phosphor materials can be embedded in different types of polymers. The results can be summarized as follows.

Effect of Eu and Y concentrations in ZrO₂ crystals

The ZrO₂ crystal co-doped with Y and Eu was obtained under calcination at 800 °C for 1 h. The doping of ZrO₂ crystal with Y and Eu leads to the phase transformation. The monoclinic phase of pure ZrO₂ crystal transforms to the tetragonal and/or the cubic phase, depending on the concentration of Y and Eu. The morphology of Eu and Y doped ZrO₂ crystal also exhibits irregular shape particles with dimension comparable to that of the pure ZrO₂. The excitation spectra by monitoring the emission at 610 nm suggest that most of the Eu³⁺ ions are substituted at Zr⁴⁺ sites in the ZrO₂ lattice. PL spectra of Eu and Y doped ZrO₂ crystals under the UV excitation at 260 nm exhibit a red light emission. We suggest that the phase transformation is the main reason for the increase of PL intensity of Eu doped ZrO₂: 7%Y crystals. The PL intensity reached maximum at 3 mol% of Eu. When the mole ratio of Y was varied, the ZrO₂: 4%Y: 3%Eu crystal exhibited highest luminescence efficiency with cubic structure. These results indicate that the content of Eu and Y in the crystals affects the luminescence efficiency.

Effect of calcination temperatures

The synthesis of our samples in this study requires the calcinations at high temperature to induce the crystallization of the materials. The calcination temperatures were varied from 600 to 1000 °C to trigger the crystallization process of ZrO₂: 4%Y: 3%Eu. All samples exhibit a cubic phase. The increase of calcinations temperature from 600 to 800 °C hardly affects the crystal size. All particles exhibit irregular shape. When calcination temperature was increased above 800 °C, the crystal size became

larger. The PL intensity varied with calcination temperature. The calcination of sample at 800 °C provided highest PL intensity. This result indicates that the calcination temperature affects the de-excitation process of the crystals.

Effect of chelating agents

The complexation of chelating agents with metal ions of the precursors was required in this study. We investigate four types of chelating agents including EDTA, citric acid, malic acid and oxalic acid. These chelating agents have different number of carboxylic group, which is a major functional group for the complexation with the metal ions. The variation of chelating agents does not affect the crystal structure of ZrO_2 : 3%Eu: 4%Y. The morphologies of all samples exhibit the densely packed particles. The structure of chelating agents hardly affects the morphology of samples. The PL spectra of all samples exhibit a red luminescent emission. The use of oxalic acid as chelating agent yields the ZrO_2 : 3%Eu: 4%Y crystal with highest luminescence efficiency.

Preparation of ZrO_2 : Y: Eu in polymeric composite films

In our earlier sections, the ZrO_2 : 4%Y: 3%Eu crystal can emit a red light and exhibit highest luminescence. We embed this material into polymeric films by using spin casting method. The composite films are smoother and thinner than the films fabricated from drop casting method. The PL emission spectra of composite PVA films mixed with various concentrations of ZrO_2 : 4%Y: 3%Eu ranging from 0.05 to 2 wt% exhibit a red luminescent emission. The emission intensity of composite films systematically increases with increasing concentration of ZrO_2 : 4%Y: 3%Eu. The results indicate that ZrO_2 : 4%Y: 3%Eu powder can be utilized as a composite PVA film. The composite films of ZrO_2 : 4%Y: 3%Eu embedded in different polymers are also prepared by using spin casting method. The polymers included PS, PMMA and PAA. PL spectra of composite films of ZrO_2 : 4%Y: 3%Eu embedded in different polymers exhibit strong red region. The highest emission intensity of composite films is obtained from ZrO_2 : 4%Y: 3%Eu embedded in PS. The polarity of polymer probably affects the luminescence efficiency of materials.