A Study of Small Particle Yttrium Oxide Type Phosphors prepared from Solution using a Sacrificial Micellar Phase as a Combustion Fuel

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INTRODUCTION

In 1999 the facile self-assembly of the red emitting phosphor yttrium oxide europium $(Y_2O_3:Eu^{3+})$ from solution using a sacrificial micellar phase was reported. The micellar phase was assembled using the alkyl ammonium salt $(C_{12}H_{25}NH_3)Cl$ in an ethanolic solution. The resulting fine powder had smaller particles, ranging in size from 0.1 to 1.0 μ m, than the commercial cubic $Y_2O_3:Eu^{3+}$ phosphor.¹ In this work we used a longer chained aliphatic amine, $C_{16}H_{33}NH_2$, as fuel.

RESULTS AND DISCUSSION

A typical preparation was carried out using the following method. A warm ethanolic solution of YCl₃ (55.37 g, in 25 ml, 0.25M) and EuCl₃ (1.76 g in 25ml, 0.02M) was added to a warm solution of $C_{16}H_{33}NH_3Cl$ in ethanol. This gave a stoichiometric ratio of 1:1 for the combined metal chloride to alkylammonium chloride present in the solution. In other preparations the metal chloride to alkylammonium chloride ratio was 1:3. The solution was heated on a hot plate to about 100°C where the initial yellow colour disappeared and the solution was reduced in volume until it became viscous and a pale yellow mass was then apparent. A portion of each product was then fired in a furnace (wherein the temperature was either set at 650°C or 900°C) for 30 min. This allowed the combustion of the alkylammonium chains.

For the samples that were produced at the furnace set temperature of 650°C, there was no evidence for cubic $Y_2O_3:Eu^{3+}$. On the other hand, the samples that were produced at the furnace set temperature of 900°C showed the presence of crystalline material and they showed a Raman spectrum and X-ray powder diffraction pattern identified as cubic $Y_2O_3:Eu^{3+}$.



FIGURE 1. Raman spectra of phosphor samples prepared at metal chloride to alkylammonium chloride ratios of:- (a) 1:1 and fired at 650°C, (b) 1:3 and fired at 650°C, (c) 1:1 and fired at 900°C, and (d) 1:3 and fired at 900°C. The exciting wavelength was equal to 632.8 nm.

As can be seen from the Raman spectra shown in Figure 1, a strong Raman band at 377 cm^{-1} (arrowed in Figure 1) is absent when the phosphor nanoparticles are fired at 650° C (see Figures 1a and 1b), but appears when the phosphor nanoparticles are fired at 900° C (see Figures 1c and 1d). This band is due to the cubic phase of Y₂O₃:Eu. In addition, a number of other strong bands also appear in Figures 1c and 1d; these are due to the photoluminescence of the Y₂O₃:Eu phosphors under 632.8 nm excitation.

In conclusion, when the oven temperature was set at 900°C (but not at 650°C), the combustion fuel present in the samples was sufficient to raise the temperature over 900°C for a long enough time period for nanometer sized crystallites of the cubic Y_2O_3 :Eu³⁺ phase to form.

REFERENCES

1. T.G. Ireland, J, Silver, C. Gibbons and A. Vecht, Electrochem.and Solid State Letts., 2, 52-54, (1999).