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Development of coating for temperature sensing

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Abstract

Fluorescence from a phosphor coating will indicate the temperature of the substrate to which it is attached, assuming thermal equilibrium is achieved. Synthesis of a phosphor pigment with europium as a doping element in RE_2O_3 (RE = Y) has been made. The synthesis is carried out by direct combustion method. The prepared Y_2O_3:Eu phosphor pigment was characterized by XRD, SEM and TEM. Using the synthesized phosphor pigment the paint has been formulated in silicone resin. The fluorescence was measured using fluorescence spectrometer and results are presented in this paper.

Keywords: Phosphor; Fluorescence; Silicone resin; Temperature sensitive paint

1. Introduction

For many years, phosphor thermometry has been used for non-contact measurements in hostile high-temperature environments. Fluorescent materials are used as powders, either suspended in binders and applied as paint or applied as high-temperature sprays. Phosphors are fine powders that are doped with trace elements that will emit visible light when suitably excited. Many of them are ceramic powders and they can withstand extremely high temperatures. A phosphor coating can indicate the temperature of the surface on which it is applied as a thin coating since the fluorescence characteristics of the phosphor pigment changes with temperature [1–7]. Y_2O_3 is an excellent substrate for phosphors because of its chemical stability. The Y_2O_3:Eu is an efficient red-emission phosphor. Traditionally Y_2O_3:Eu phosphor is prepared by high-temperature solid-state reaction at 1400–1500 °C for several hours [8,9] The phosphor particle synthesized by this method is massive and the grain size is very big and must be ground or milled to get finer powder. Jing et al. [9] have described the preparation of spherical phosphor (Y_2O_3:Eu) particles that are mono-sized for given set of conditions. They have used urea homogenous precipitation method [10–13]. They have reported that by modifying the standard urea homogenous precipitation method, spherical Y_2O_3:Eu phosphors can be prepared and the luminescence increases with the particle size of the prepared phosphor. Ungur et al. [14] have described and prepared Y_2O_3:Eu by thermal synthesis at 1100–1400 °C from homogeneous mixtures of yttrium oxide, Eu_2O_3 and alkaline earth salts as flux. They have concluded that to obtain efficient Y_2O_3:Eu, flux nature and concentration, firing temperature and activator quantity have to be carefully chosen. Zhang et al. [15] have synthesized nanometer Y_2O_3:Eu by a simple sol–gel process using citric acid as chelating agent. The phosphor was characterized by XRD, TEM and spectroflurometer. The synthesized phosphor was calcined from 500 to 1250 °C. They have calculated the crystal size using the Scherrer equation. By using a simple sol–gel process they have synthesized nanometer scale phosphor. The luminescence intensity has found to be increased.

2. Experimental

2.1. Synthesis of Y_2O_3:Eu phosphor pigment

TheY_2O_3:Eu nano-phosphor pigments were prepared by the method described by Bolstad and co-workers [16] by direct combustion synthesis using urea as a fuel. The mixed nitrate solutions were preheated to 500 °C for 10 min to get a puffy white mass. This mass was sintered at 850 °C for overnight. The dopant concentration is varied between 0.6, 1.2 and 2.4% atomic weight in the yttrium host lattice.

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2.2. Characterization of the Y_2O_3:Eu phosphor

The prepared phosphor materials were analyzed by XRD, SEM, TEM and fluorescence.

2.2.1. XRD analysis

The X-ray diffraction pattern of Y_2O_3:Eu pigment powder was taken with analytical (Model PW3040/60) X-ray diffractometer using Cu Kα radiation in the 2θ range 5–75° at the scan rage of 0.0170° 2θ with continuous scan type with scan step time of 15.5056 s.

2.2.2. SEM analysis

The scanning electron micrograph of Y_2O_3:Eu pigment was analyzed using Hitachi (Model S3000 H) instrument. The pigment was spread over a copper block over which gold was sputtered.

2.2.3. TEM analysis

TEM analysis of the developed phosphor is done in order to get accurate data of the grain size of the Y_2O_3:Eu phosphor using JEOL model TEM 1200EX.

2.2.4. Fluorescence measurements

The fluorescence of the prepared pigment powder sample was measured using Fluromax-3 spectrofluorometer (HORIBA-JOBIN YVON Inc.). Optimization of dopant content in the prepared Y_2O_3:Eu pigment was done by measuring the fluorescence spectra of the phosphor samples by exciting them with 262 nm blue light.

2.3. Paint formulation

Commercial silicone resin SILRES KX was obtained from M/S WACKER METROARK SILICONES. The solid content of the resin is 50 ± 1, viscosity is 6–12 mm^2/s, density is 1.05 g/cm^3 and the flash point of the resin is 24 °C. The developed phosphor is used as pigment in silicone resin by dispersing 10% by weight. Xylene was used as solvent. The paint is applied over quartz tiles of 2 cm × 2 cm size by airbrush to get dry film thickness of 20 ± 2 μm. The painted samples were cured at room temperature for 24 h.

2.4. Testing of temperature sensing paint (TSP) at different temperatures

Fluorescence measurements were carried out at room temperature using Fluromax-3 spectrometer. The excitation wave length was fixed at 262 nm and the fluorescence response of the TSP was recorded by heating the quartz samples from room temperature to 400 °C at every 100 °C. A mini furnace has been designed and fabricated to measure the fluorescence of the developed temperature sensitive paint. The temperature of this furnace is controlled manually and the accuracy of this setup is ±5 °C. Fig. 1 shows the furnace setup. The painted samples are kept in this setup and the fluorescence of the samples was measured using optical fiber bundle which was supplied with Fluromax spectrometer.

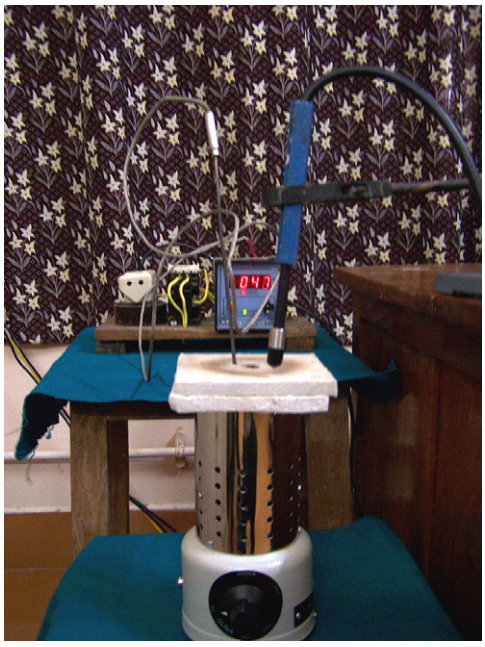

Fig. 1. Furnace setup.

3. Results and discussion

Fig. 2 shows the XRD pattern of the synthesized Y_2O_3:Eu phosphor. The XRD data of the prepared samples have matched well with PCPDF DATA FILE 25-1011. Thus the composition of the prepared sample is found to be $(Y0.95Eu\ 0.05)_2O_3$. The particle size of the sample is calculated by Scherer and Williamson Hall method and found to be of the order of 50 nm, indicating that the phosphor prepared is of nano-size. Fig. 3 shows the SEM image of Y_2O_3:Eu phosphor and it can be seen that the particles have a spherical shape. The TEM image is given in Fig. 4. The grain size of Y_2O_3:Eu phosphor sintered at 850 °C is in the range of 50–55 nm which is in agreement with the particle size obtained from XRD data. Fig. 5 shows the fluorescence spec-

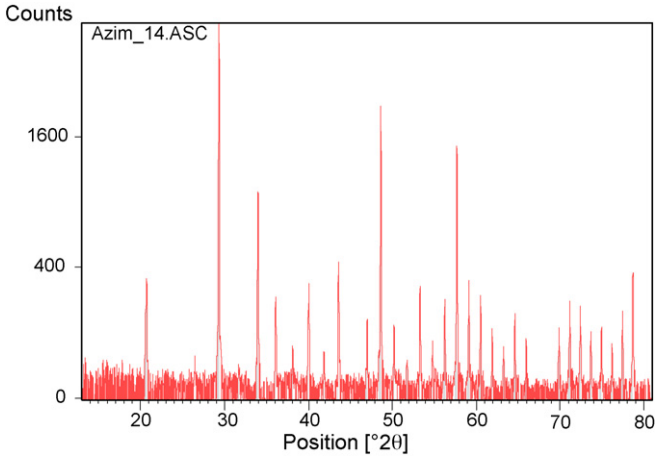

Fig. 2. XRD pattern of Y_2O_3:Eu phosphor.

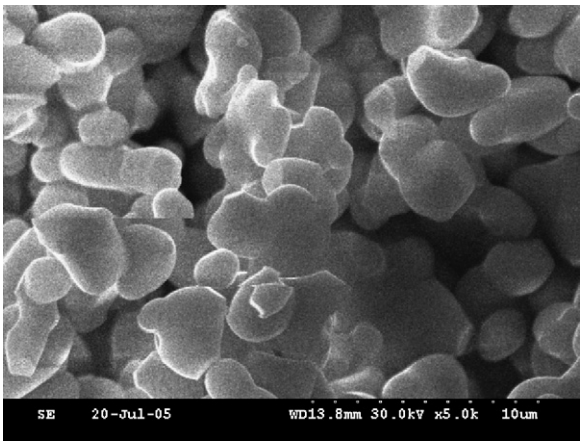

Fig. 3. SEM Micrograph of Y_2O_3:Eu powder .

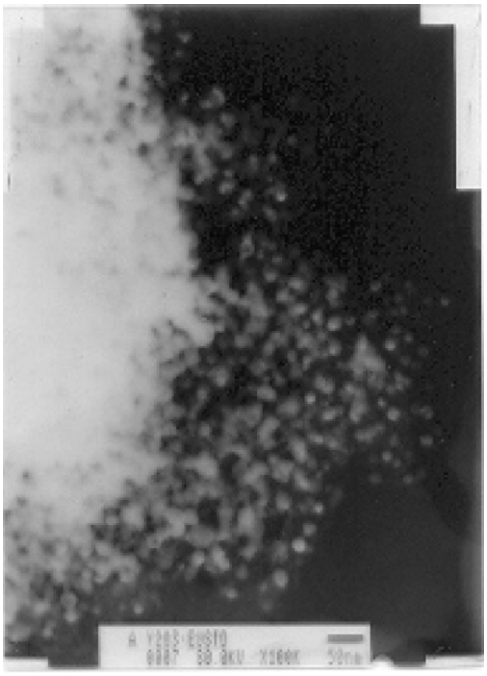

Fig. 4. TEM image of Y_2O_3:Eu powder.

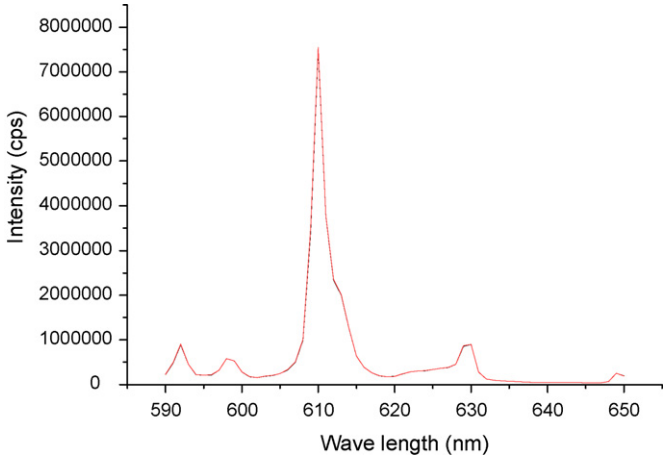

Fig. 5. Fluorescence spectra of Y_2O_3:Eu powder.

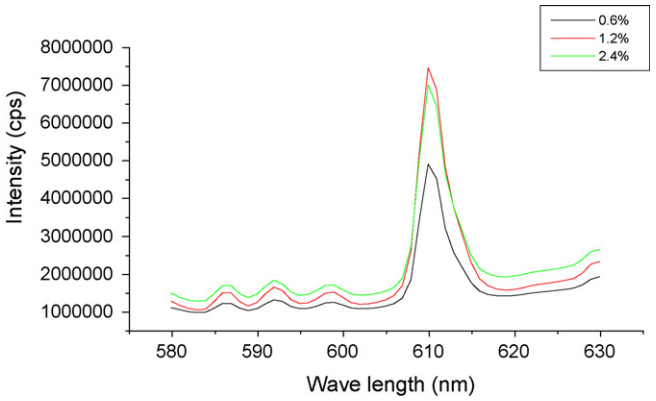

Fig. 6. Optimization dopant content.

tra of Y_2O_3:Eu powder pigment sample at room temperature. The prepared Y_2O_3:Eu sample has a maximum emission peak at 610 nm when excited with 262 nm UV light which is well in agreement with the reported literature. Optimization of the dopant Eu content was done by measuring the fluorescence of the powder samples by exciting with 262 nm UV light. The fluorescence spectra for various dopant levels of Y_2O_3:Eu pigment samples are shown in Fig. 6. From this, it can be concluded that at a concentration of 1.2% by atomic weight the fluorescence emission was maximum and hence the dopant content was optimized at 1.2%.

The pigment content in the paint is optimized by loading different wt% of pigment from10 to 20% and comparing the fluorescence spectra at room temperature. The results are shown in Fig. 7. It can be seen that maximum fluorescence emission intensity is obtained for 12% loading of Y_2O_3:Eu phosphor pigment in the paint. Hence for further studies TSP'S with 12% pigment loading have been made.

The fluorescence spectrum of TSP with 12% pigment loading was subjected to heating and the fluorescence response of the paint was recorded at room temperature 30, 350 and 400 °C. The results are shown in Fig. 8. The paint sample was found to emit fluorescence up to 400 °C. After 400 °C, the binder becomes brittle; hence fluorescence measurements were not possible. Fig. 9

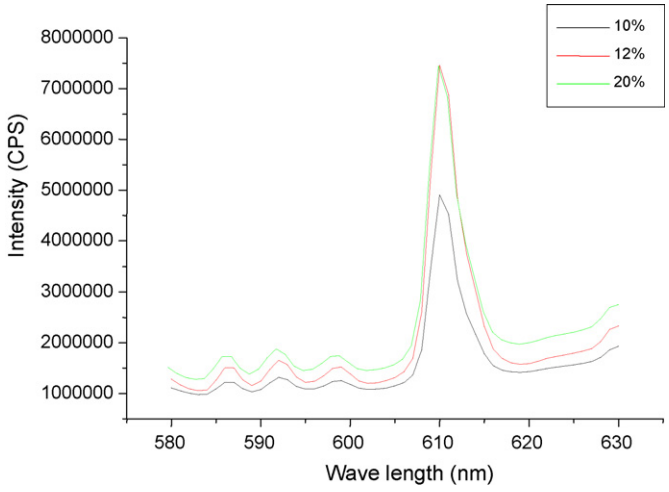

Fig. 7. Fluorescence spectra of Y_2O_3:Eu paint with different pigment loading.

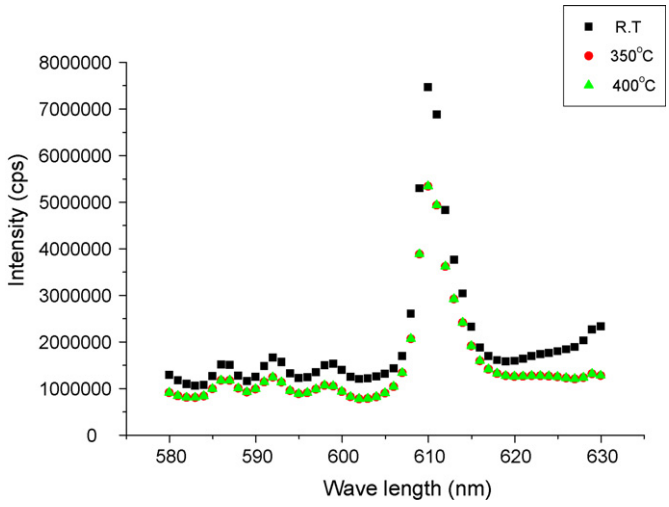

Fig. 8. Fluorescence spectra of Y_2O_3:Eu paint (12% loading) at various temperatures.

Fig. 9. Painted quartz tile at 450 °C.

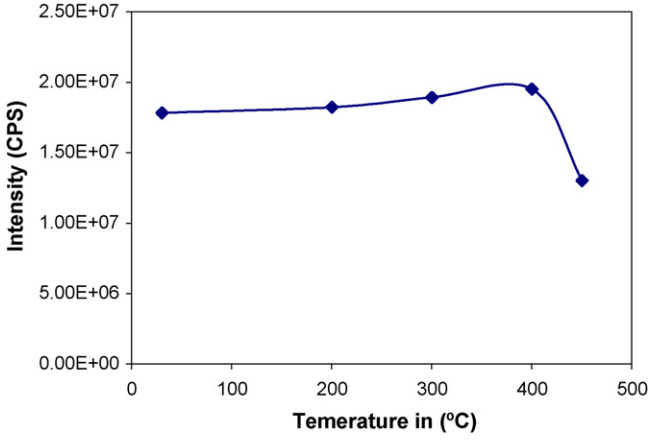

Fig. 10. Peak fluorescence intensity at 611 nm for various temperatures.

shows the photograph of the TSP coated quartz tile heated upto 450 °C.

Fig. 10 shows the peak emission intensity with temperatures. From this, it can be concluded that there is a slight increase in the emission intensity as the temperature is increased. The synthesized Y_2O_3:Eu phosphor has measurable fluorescence upto 1000–1100 °C as reported in literature. In the present study the fall in fluorescence above 400 °C is due to the disintegration of the silicone binder used in the paint formulation. Further studies with ceramic binders which can withstand temperatures upto 1000 °C are in progress.

4. Conclusions

Phosphor doped with trace element can be used as a pigment in formulating temperature sensing paint. When suitably excited, these phosphor pigments are found to emit visible light. By measuring the fluorescence response of the phosphor pigment it is possible to determine the temperature of the substrate. The drop in fluorescence intensity after 400 °C is due to the degradation of the silicone binder used in the study.

Acknowledgements

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