Synthesis and Characterisation of Y_2O_3:Eu/Ag powders

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Abstract

Luminescent materials have a wide range of applications, such as organic light emitting devices, inorganic light emitting diode devices, displays, bio detections, etc. In this research, we showed the synthesis of Y_2O_3 (core): Eu (dopant)/Ag (shell) powders through Ultrasonic Spray Pyrolysis in scale up conditions at 800 °C with a flow rate of air and at constant concentration of yttrium nitrate, europium nitrate and silver nitrate according the previously performed experiments. Detailed characterisation was performed on the produced Y_2O_3:Eu/Ag powder using STEM, SEM and EDX analysis. Microstructure observation showed that Y_2O_3:Eu/Ag powders are spherical, containing 50 % yttrium oxide, 45.2 silver and 0.65 europium.

Introduction

Luminescence promotes emission of light from a material at a certain excitation wavelength. It can be categorised into diverse kinds, depending upon the various involved processes of excitation. The luminescent materials were prepared using the sol-gel and self-propagating high temperature synthesis routes [1]. The effects were studied of thermal treatment and dopant concentration on the phase composition, crystallite size, lattice parameters and luminescent properties of the synthetised phosphors [2].

Ag@$(Y_{0.95}Eu_{0.05})_2O_3$ nanocomposites were synthesised by single step Ultrasonic Spray Pyrolysis (USP) at 800 °C using 1.5 l/ min air by Alkan et al. [3]. A detailed parametric study was conducted on samples with varying silver contents and heat treatment conditions. The effect of silver in both as prepared and heat-treated samples were elucidated in terms of structural and functional properties. 2.5 wt% Ag incorporation followed by 2 h heat treatment at 1000 °C is reported as the most promising red light emitting phosphor synthesis conditions via USP.

Deep insight into the photoluminescent monocrystalline particles was reported by Alkan et al. [4]. The red light emitting down-converting Ag@Y_2O_3:Eu^{3+} phosphor particles were synthesised by one-step Ultrasonic Spray Pyrolysis and further exposed to the heat treatment at 1000 °C (12 h). A detailed investigation of the structural and

functional properties of the as-prepared and heat treated particles was conducted in a comparative manner. This comparative study implies a good correlation between the mechanical and luminescence behaviour of phosphors, both strongly influenced by the particles` structural properties.
Alkan et al. [5] have reported that reproducible, spherical and dense nano/submicron silver nanoparticles have been synthesised and collected successfully with this scale up USP equipment. From a general point of view, the performance of the scaled up Ultrasonic Spray Pyrolysis device is satisfactory. The demo equipment, which is devoted at the beginning to silver based nano/micro structures, was firstly tested with a series of 36 hours of continuous experiments [5].
Our aim is scale up of the synthesis of the Y_2O_3:Eu/Ag powders by Ultrasonic Spray Pyrolysis and characterisation of the obtained powders.

Experimental

Material and Procedure

The synthetic water solutions of yttrium nitrate, silver nitrate and europium nitrate were used for the synthesis of Y_2O_3:Eu/Ag powders using the Ultrasonic Spray Pyrolysis method, as shown in Figure 1.

Fig. 1. Ultrasonic Spray Pyrolysis scale up equipment

As represented in Figure 1, the scale up equipment has been designed with five main parts, consisting of an ultrasound generator, gas system, heating/reaction furnace and wet scrubbing system. There are five ultrasound generators, PRIZnano, Kragujevac, Serbia (2.5 MHz), which are regulated automatically and allow running a continuous process. A gas system with controlled volume resp. mass flow of pure and mixed gases allows for carrying aerosols produced by the ultrasound generators to the heating zones. Each aerosol generator is connected to an individual reaction tube. These five reaction tubes are located in a wall heated furnace with four separately regulated heating zones (max. 1000 °C). At the end of the reaction zones, the stream containing the carrier gas and nanoparticles is carried to the powder collection area. There is one wet scrubber system. The produced aerosols of yttrium nitrate, silver nitrate and europium nitrate are transported to the reaction tubes at 800°C using air. After thermal decomposition and formation of Y_2O_3:Eu/Ag powder, the particles were collected by the wet scrubber. The morphological analysis of the formed particles was performed using SEM and EDS Analysis.

RESULTS and DISCUSSION

ICP-OES analysis of the obtained powder at the IME, RWTH, has shown the presence of metals (weight %): 43.9 Ag, 39.8 Y and 0.6 Eu. The morphological characteristics are shown in Figure 1:

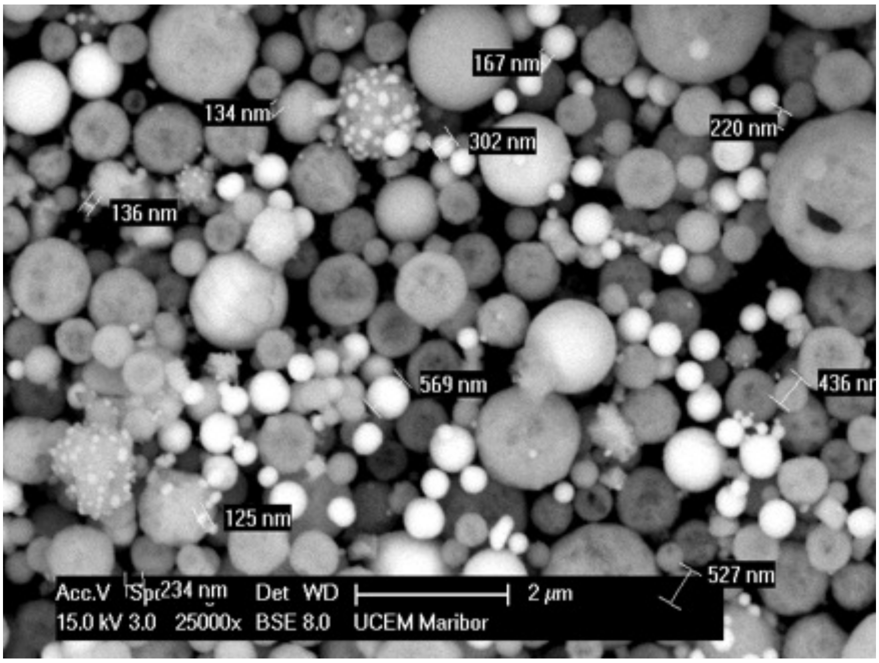

Fig. 1. SEM analysis of Y_2O_3:Eu/Ag powders obtained by Ultrasonic Spray Pyrolysis in scale up conditions (Ag-white; Y_2O_3-grey, Eu-present in minor quantities)

The particles` sizes were between 100 and 600 nm. The qualitative and quantitative EDS analysis is presented in Figure 2.

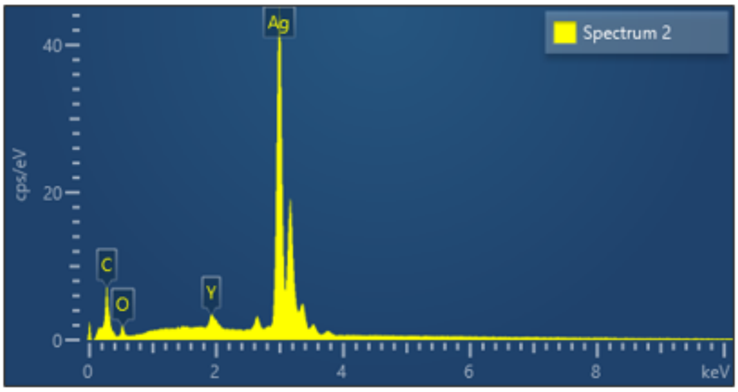

Figure 2: Qualitative EDS Analysis of the obtained Particles

One detailed SEM and quantitative EDS-analysis is shown in Figure 3 and Table 1, where primary and secondary particles are presented.

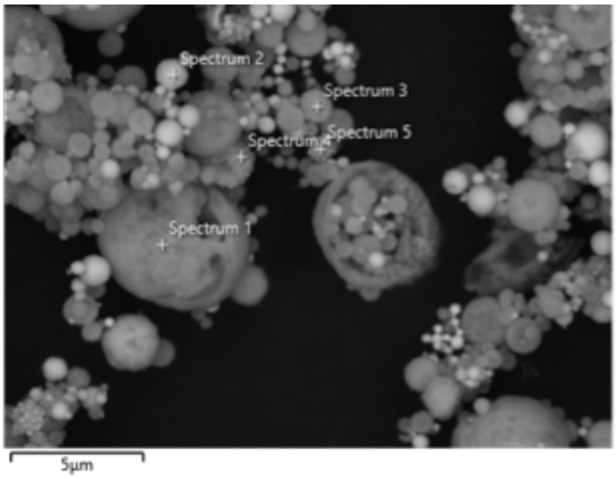

Figure 3: SEM and EDS Analysis of particles

Spectrum	O	Y	Ag	Eu	Total
Spectrum 1	30.19	62.36	6.21	1.23	100.00
Spectrum 2	23.47	57.39	17.58	1.56	100.00
Spectrum 3	23.59	52.94	21.80	1.67	100.00
Spectrum 4	14.95	9.69	75.36		100.00
Spectrum 5	16.60	20.13	63.27		100.00
Spectrum 6	29.62	52.92	16.78	0.68	100.00
Spectrum 7	30.53	55.63	13.06	0.78	100.00

Table 1: Quantitative EDS Analysis of the Powder

Quantitative analysis in Table 1, has confirmed the different chemical content of the metals.

CONCLUSION

The synthesis of Y_2O_3 (core): Eu (dopant)/Ag (shell) powders (43.9 Ag, 39.8 Y and 0.6 Eu) was performed successfully through Ultrasonic Spray Pyrolysis in scale up conditions at 800 °C with a flow rate of air, and at constant concentration of yttrium nitrate, europium nitrate and silver nitrate according the previously performed experiments in laboratory conditions. The obtained particles are spherical with different sizes between 100 und 600 nm. This powder was tested for 3D printing in order to establish whether the produced nanosized particles are suitable for this application.

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