личину сенсорного отклика, однако в литературе имеется предположение о более значительной роли дефектности кристаллов и состояния поверхности (наличие поверхностных ОН-групп) по сравнению с другими факторами.

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УДК 667.7 + 681.7

SONOCHEMICAL SYNTHESIS OF $(Y,Eu)_2O_3$ PHOSPHORS

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Y_2O_3:Eu^{3+} phosphor exhibits red emissions and possesses excellent chemical stability. It is the only existing red phosphor used in three band-fluorescent lamps. Y_2O_3:Eu^{3+} has attracted great interest for use as red phosphor in fluorescent lamps, high-resolution projection TVs, protection devices and low voltage displays, being a component of cathode ray tube, plasma display panels and field emission displays [1, 2]. High refractive index (>1.9), large band gap (5.8 eV), physical and chemical stability of pure or doped Y_2O_3 provide its potential application in optoelectronics. The morphology and the particle size affect the emission intensity of phosphor. In general, the luminous efficiency of phosphor reduces with decreasing particle size until the quantum size effect does not occur.

Various synthesis methods of the phosphor are studied in order to obtain nanocrystalline materials. Reducing the particle size of the phosphor will expand fields of its application. For instance, nanocrystalline phosphors are suitable for high definition television (HDTV) where conventional bulk phosphor cannot be used [1].

Experimental. In this paper, the structural and morphological properties of samples prepared by the sol-gel and the ultrasonic methods were compared. In the first case, an aqueous solution of ammonia (9.24 M) was added dropwise to an aqueous solution of yttrium and europium nitrates (0.45 M). The process was carried out under continuously magnetic stirring. The resulting sol was decanted, then dried in air at 150 °C. The xerogel was calcinated for 2 h at various temperatures (400, 600, 800 °C). In case of the ultrasonic synthesis method, the mixture of reagents was exposed to ultrasound treatment with a frequency of 29 kHz.

Thermal behavior of the xerogels was evaluated by simultaneous thermal gravimetric analysis DTA–TG (NETZSCH STA 449 F3 Jupiter) from 20 to 600 °C in nitrogen with heating rate of 10 °C/min. X-ray powder diffraction (XRD) analysis was carried out with DRON-3 X-ray diffractometer with $CuK\alpha_1$ radiation (α = 1.5405 ˚A). The reference data were used from the PDF2 database. The average crystallite size (D) was determined from the diffraction peak broadening using the Scherrer formula $D = K\lambda/\beta\cos\theta$, where D is the average particle size in (nm), K is the Scherrer constant (0.89), λ is the wavelength of X-ray source, β is the full width at half-maximum and θ is the Bragg's angle. The experimental XRD data were processed with HighScorePlus software. Fourier-transform infrared (FTIR) spectra were recorded on Avatar 330 FTIR spectrometer equipped with Smart Diffuse Reflectance system at room temperature using KBr pellet method.

Electron microscopy study was performed by means of the scanning electron microscope Leo-1420 Carl Zeiss.

Results and discussion. Intensive luminescence in the red region of the spectrum under UV excitation (270 nm) was acquired after xerogel calcination at ~550 °C (2 h) and higher.

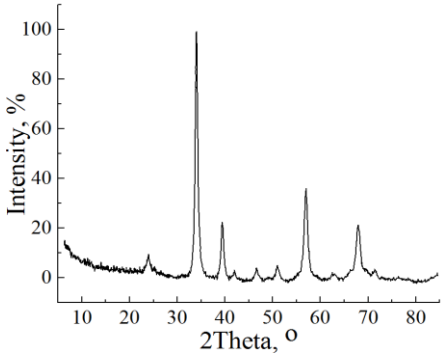

Fig. 1 – XRD spectrum of the sample (powder) of the composition Y_2O_3–Eu_2O_3 (4 % wt. Eu_2O_3, annealing 600 ˚C, 2 h). prepared by the sonochemical method

XRD analysis established the formation of a solid solution $(Eu,Y)_2O_3$ based on the cubic structure of Y_2O_3. Figure 1 shows XRD spectrum of the sample prepared by the ultrasonic method (sol-gel synthesis in an ultrasonic field).

The sizes of crystallites for powder samples, synthesized under ultrasound and annealed at 400, 600 and 800 C, were 7, 10 and 13 nm, respectively. In the absence of an ultrasonic field, the indicated values were 8, 12 and 16 nm. In the process of synthesis, the same phase composition was formed over the temperature range of 400 ÷ 800 °C, *i. e*. cubic modification of yttrium oxide Y_2O_3 (Card № 43-1036).

According to TGA data (Fig. 2) complete decomposition of $Y(OH)_3$ and $Eu(OH)_3$ took place at calcination temperature 600 °C in air. Weight loss of sample after six steps of physical and chemical dehydration was 83.51 %.

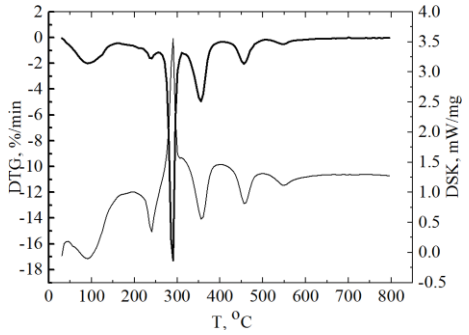

Fig. 2 – DTA–TG spectrum of the sample of the composition Y_2O_3–Eu_2O_3 (4 % wt. Eu_2O_3, annealing 600 ˚C, 2 h) prepared by the sonochemical method

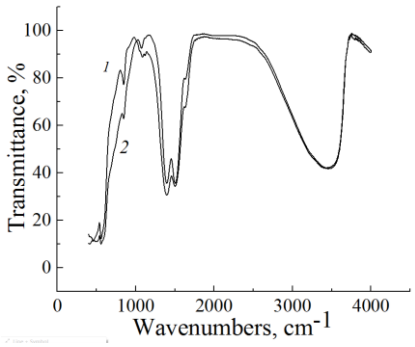

Fig. 3 – FTIR spectra of the samples of the composition Y_2O_3–Eu_2O_3 (4 %wt. Eu_2O_3, annealing 600 ˚C, 2 h) synthesized by sol gel technique: 1 - in an ultrasonic field of 29 kHz; 2 - without ultrasonic treatment

The sonochemically derived as-prepared $Y(OH)_3$:Eu (4 %wt) show OH bands at 3615 cm^{-1} along with bands near 475 ÷ 720 cm^{-1} of Y–O, Eu–O that are in agreement with literature data. After calcination at 800 °C, the FTIR spectra containY–O and Eu–O adsorption bands at 410,2 ÷ 602,8 cm^{-1}. An insufficient difference in the absorption intensity of the denoted areas was observed for the samples, prepared with the application of ultrasonic field and without it, that could be induced only by difference in particle size and morphology of the powders (Fig. 3).

The broad absorption band with a maximum at 3650 cm^{-1} is probably associated with fluctuations of physically and chemically bound water, the intensity of this band decreases with an increase of calcination temperature.

According to SEM data, $Y(OH)_3$:Eu powder (4 %wt) (Figure 4) is a mixture of nanoscale particles of irregular shape with the size of 50÷ 150 nm. The powder consists of fairly homogeneous particles, partially agglomerated. The proportion of particles with the size less than 250 nm is 708.2 (per 1000 particles), their density is ≈ 5.58 μm^{-1}. Within the 0-250 nm particle fraction, irregularly shaped particles from 50 nm in diameter are distinguishable. Separate large particles up to 2.0 microns are agglomerates of flattened particles.

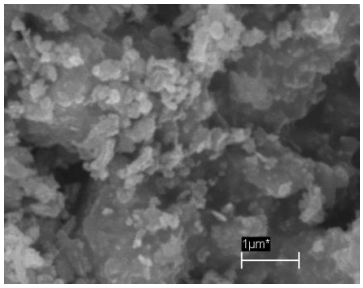

Fig. 4 – SEM imageof the Y_2O_3–Eu_2O_3 (4 % wt. Eu_2O_3, annealing 600 °C, 2 h)

Conclusion. The use of the ultrasonic technique provided a significant decrease in the size of crystallites (areas of coherent scattering) and the size of separate particles.

In comparison to the Pechini method, which allows to obtain particles with crystalline size 21 nm [3] after annealing at 600 °C, the sol-gel method with ultrasonic treatment is notable for its simplicity and environmental friendliness (there is no emission of gases during the degradation of the organo-containing gel, duration of the synthesis process is much shorter).

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