

Research Article

Rare-Earth Doped Nanocrystalline Phosphors for Field Emission Displays

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The cathodoluminescence properties of rare-earth (RE = Ce, Eu, Tb) doped nanocrystalline phosphors (Y_2O_3 , $Y_3Al_5O_{12}$) were investigated. Their structure and morphology were determined and correlated with optical properties. The effect of grain sizes on emission yield of RE doped nanophosphors has been investigated. A possibility of application of RE doped nanophosphors for efficient field emission display (FED) devices has been discussed.

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1. INTRODUCTION

Phosphors are efficient luminescent materials and irreplaceable components of light-emitting devices like cathode ray tubes (CRTs), plasma display panels (PDPs), and field emission displays (FEDs). The last ones have gained a great interest [1] and have been recognized as one of the most promising technologies in the flat panel displays (FPDs) market [2–6] due to their most important features like great brightness, wide horizontal and vertical view angles, good contrast ratio, high efficiency with a low power consumption, short response time, and wide work temperature range. The PDPs in contrast to the liquid crystal displays (LCDs) [7] are characterized by low contrast ratio, low view angle, and narrow operating temperature range, but they have low efficiency and high power consumption resulting in overwarming during work [6, 8, 9]. Such behaviors do not appear in FEDs at all. Additionally, FEDs have high brightness (3000 cd/m^2) and very high efficiency ($2 \text{ W/10.4}''$). The only problem that appears in FEDs is that the operating time is shorter than in the case of other described technologies. This problem is connected with progressive degradation of vacuum level in the display space [10–13]. There are three main sources of gasses in FEDs. The first one is the gas emitted during device encapsulation process [11]. The second source of gasses is the employment of inappropriate materials (e.g., porous glasses) in the device construction [10, 13]. The third and most important source of gasses is improper type of phosphors [6, 10, 12, 13]. Examples of that class of phosphors

are semiconductors and insulators containing sulfur in the host material composition [10, 14–20], for example, ZnS , $ZnSO_4$, and $Y(\text{La,Gd})_2O_2S$. The example of the abovementioned influence of SO_2 and other gasses on FED operation has been presented in detail and discussed in work [12]. For the same reasons CdS , PbS , and $Zn(\text{Cd})S$ phosphors or even $CdSe/ZnS$ quantum dots composites need to be eliminated [6, 21]. Besides, they consist of harmful cadmium and lead which classified those materials as dangerous and disqualified from commercial application.

Other problem is a decrease of cathodoluminescence efficiency by negative loading of grain surface in phosphor layer. That is why the electrical conductivity of phosphors should be high enough to avoid charge accumulation [18, 22, 23].

The above considerations lead to a conclusion that new class of phosphors needs to be applied in field emission displays. One of the most promising solutions is rare-earth (RE) doped garnets, oxides, silicates, and some perovskites [14, 18–20, 24–26]. Unfortunately, most of them are insulators. This disadvantage could be neutralized by modulation of phosphor grain size. At present, a large majority of efforts concentrate on application of nano- and eventually submicron-size particle phosphors [27–36]. Some experimental [36, 37] and theoretical considerations [38] promote as the most efficient phosphors with submicron grain size of spherical shape [39]. However, nanoparticle phosphors could be appropriate for low voltage FED application [38]. Besides, they have a lot of other advantages and create new possibilities [16, 17, 39, 40]. In some cases, the preparation

of nanocrystalline phosphors requires much less temperature and time in comparison with conventional micro- or submicro-size grains [16, 39, 40]. Application of small grains close to spherical shape influences the efficiency of the phosphor layer and the quality of the displayed picture [39]. The employment of such materials allowed for optimal packaging of grains, avoids cutting of the picture spot and decreases scattering of emitted light in the layer [39]. Moreover, application of nanocrystalline luminescent powders allowed the fabrication of very thin light emitting film and fundamentally decreased the total resistance of phosphor layer. The thickness of the layer is connected with lower amount of gasses bound between the phosphor grains [40]. The effluent of those gasses during device operation can affect degradation of vacuum level and seriously shorten the display operating time [12, 13].

Another potential benefit connected with application of nanocrystals is a possibility of increasing conductivity of phosphors by mixing them with transparent, conductive nanocrystalline powders [41–43].

Recently, another approach towards increasing phosphor conductivity is an application of more conductive host materials like RE doped stannous oxide nanopowders [44–46]. The electrical, structural, and optical properties of SnO_2 [47–49] have focused attention on similar phosphors.

According to the above assumptions, the RE doped nanocrystalline oxides or garnets (Y_2O_3 , SnO_2 , and $\text{Y}_3\text{Al}_5\text{O}_{12}$) [50] seem to be very promising candidates for FED phosphor application. The question is about their efficiency in comparison with conventional, commercially available, micrograin size phosphors. A very important question is also the CIE (*Commission Internationale d'Eclairage*) coordinates of the phosphors. The coordinates should be suitable for application in field emission displays.

To answer these questions, we have performed the systemic studies of the nanocrystalline RE doped oxides. In particular, we have been focusing on the grain size effect on efficiency of low-voltage cathodoluminescence. We expect that the results and discussion presented in this work will contribute to the application of RE doped nanocrystalline phosphors in FED devices.

2. EXPERIMENTAL

The following nanocrystalline RE doped oxides were chosen for our studies: $\text{Eu}^{3+}:\text{Y}_2\text{O}_3$, $\text{Tb}^{3+}:\text{Y}_2\text{O}_3$, $\text{Tb}^{3+}:\text{Y}_3\text{Al}_5\text{O}_{12}$ (Tb:YAG), $\text{Ce}^{3+}:\text{YAG}$, and $\text{Tb}^{3+}:\text{YAG}/\text{Eu}^{3+}:\text{YAG}$. The phosphors have been synthesized by modified Pechini method [40, 51, 52]. The powders were sintered at different temperatures to obtain a different grain size. The structures of fabricated materials were confirmed by X-ray diffraction (XRD) by means of a stoe powder sensitive detector, filtered $\text{CuK}\alpha_1$ radiation. The average powder grain sizes were estimated from broadening of diffraction peaks, by using Scherrer equation [40, 51]. In this work, we presented the results calculated only for europium and terbium doped yttria samples (see Figure 1). The powders were then deposited by electrophoresis [53–55] on ITO glass slides [56]. As a surfactant, magnesium nitride has been used. In the last part of the procedure, the substrates were

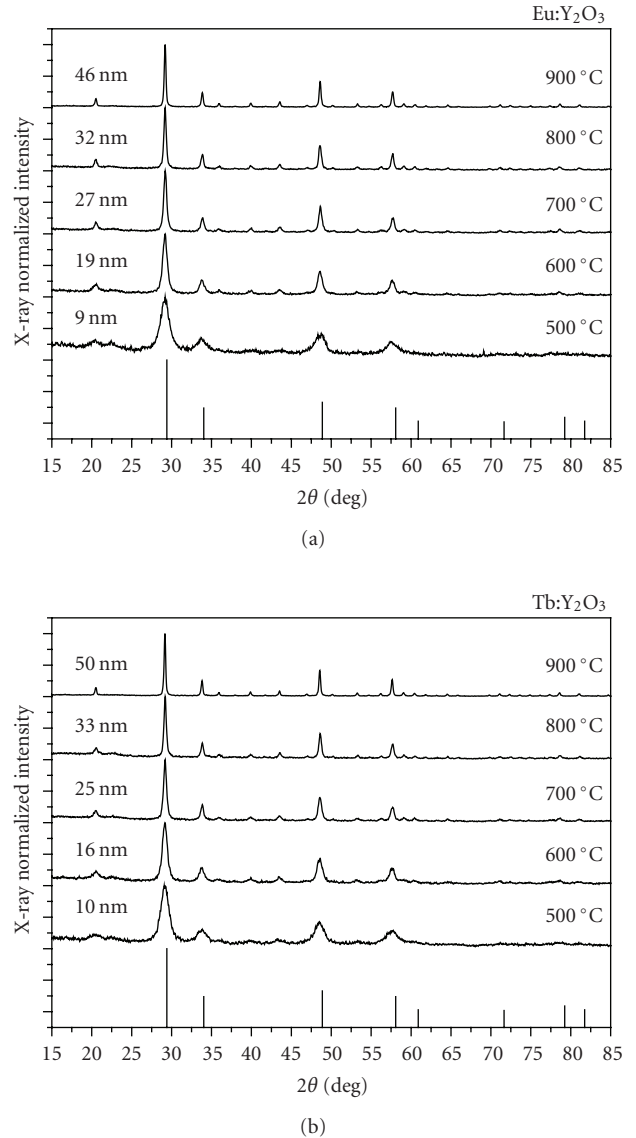


FIGURE 1: The XRD patterns of (a) $\text{Eu}:\text{Y}_2\text{O}_3$ and (b) $\text{Tb}:\text{Y}_2\text{O}_3$ nanocrystallites sintered in different temperatures.

hold in temperature of about 450°C to consolidate the layer by transforming MgOH^+ into MgO . In the future attempts, it is worth to replace $\text{Mg}(\text{NO}_3)_2$ by indium nitride or appropriate indium and tin salts.

The CL measurements [40] of the phosphor layers have been done in the setup earlier described by us [57]. The conditions of measurement device will simulate real carbon nanotubes field emission display (CNT-FED) environment. The cold cathode was made of double walled carbon nanotubes (DWNT) deposited on ITO glass slide. The emissive properties of the cathodes made in that manner were presented elsewhere [40, 57–59]. The distance between phosphor's covered anode plate and cathode plate was $250\ \mu\text{m}$ [56, 57]. The experimental conditions and procedure were similar to those described in [40, 50]. The current density of $0.9\ \text{mA}/\text{cm}^2$ for $3.2\ \text{V}/\mu\text{m}$ was registered for a diode configuration. The

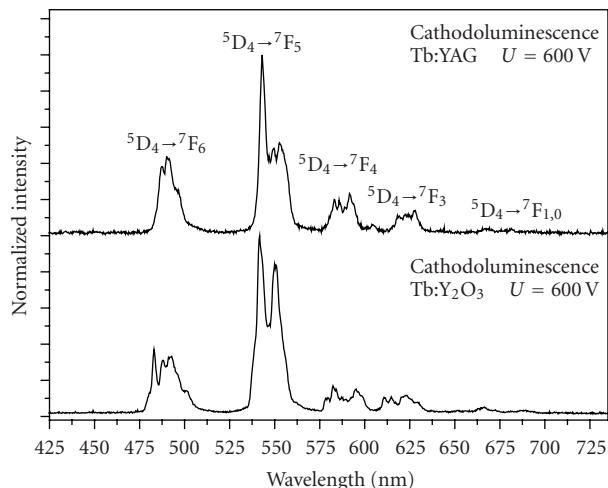


FIGURE 2: A comparison of low-voltage CL spectra registered for Tb:YAG and Tb:Y₂O₃ nanocrystallites.

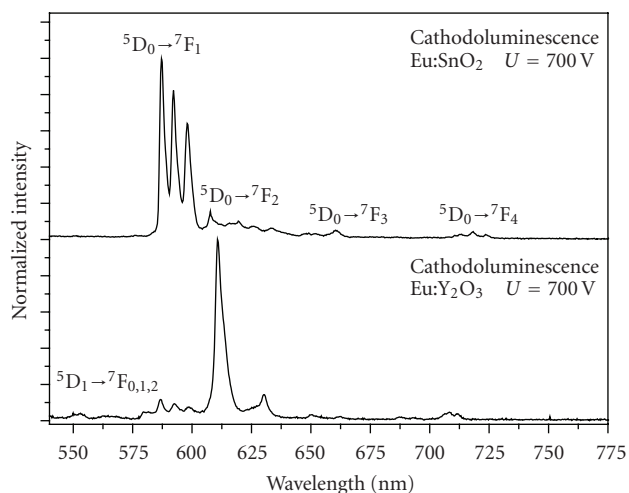


FIGURE 3: A comparison of low-voltage CL spectra registered for Eu:SnO₂ and Eu:Y₂O₃ nanocrystallites.

vacuum level in the device was determined to be 1×10^{-6} Torr. The low-voltage CL spectra (see Figures 2–5) were registered using CCD handy fiber spectrophotometer Avantes 350 with a 1000 nm spectral range and a 0.35 nm resolution.

In the second phase of the experiment, the nanopowders (Eu³⁺:Y₂O₃, Tb³⁺:Y₂O₃) were sintered at different temperatures (500°C, 600°C, 700°C, 800°C, 900°C). Then, they were mechanically mixed with a ZnS:Ag micrograin size phosphor (Phosphor Technology) and with micrograins size Cr:Al₂O₃. The micrograin size phosphors were chosen as the reference light emitting materials to study the effect of size of nanograins on output light intensity. The mass ratio of ZnS:Ag to Eu:Y₂O₃ was taken as 2:1, and in the case of Cr:Al₂O₃ to Tb:Y₂O₃, it is 4:1. The aim of this experiment was to examine the influence of grain sizes of nanopowders on intensity of luminescence by reference to the peaks connected with radiative transitions in ZnS:Ag (450 nm) and Cr:Al₂O₃

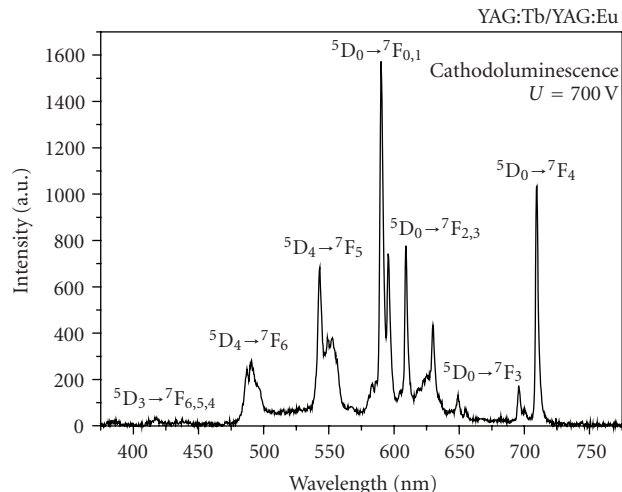


FIGURE 4: The low-voltage CL spectra of Eu:YAG/Tb:YAG blend proposed as white light emitting phosphor.

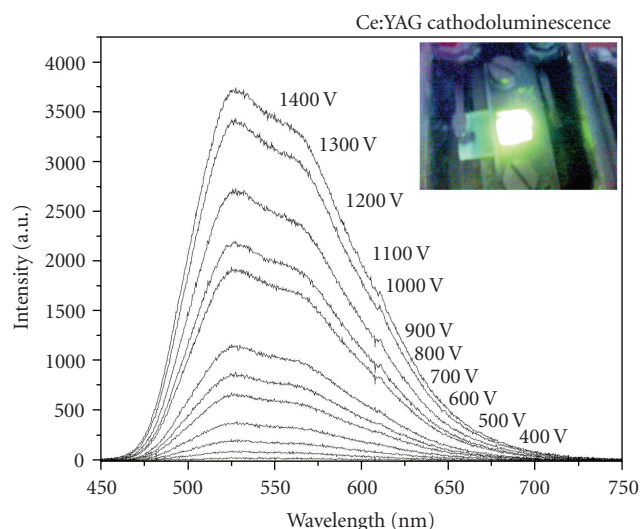


FIGURE 5: The dependence of CL spectra of Ce:YAG nanocrystals on applied voltage.

(694,1 nm). This method allowed measuring the photo- and cathodoluminescence properties. The grains size effect on photoluminescence was examined for ZnS:Ag/Eu:Y₂O₃ samples. To compare the intensity of powders with different size of nanograins, the samples with the microcrystalline powder of Eu:Y₂O₃ (Phosphor Technology) as a reference have been prepared. The medium grain size in this sample has been specified by the supplier to be 3.5 μm. The samples were excited using $\lambda_{exc} = 266$ nm of Nd:YAG laser system. The high voltage cathodoluminescence (accelerating voltage $U = 90$ kV) has been measured in specially adopted BS 500 TESLA TEM microscope chamber on Cr:Al₂O₃/Tb:Y₂O₃ electrophoretically deposited layers. The results of both experiments are depicted in Figures 6 and 7.

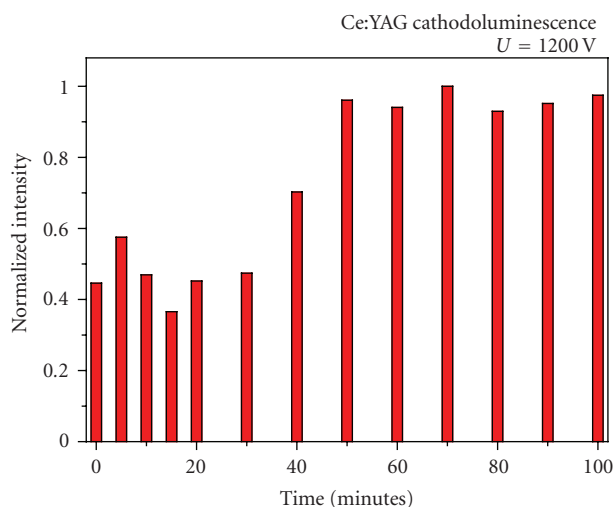


FIGURE 6: The CL time stability measured for Ce:YAG nanocrystals.

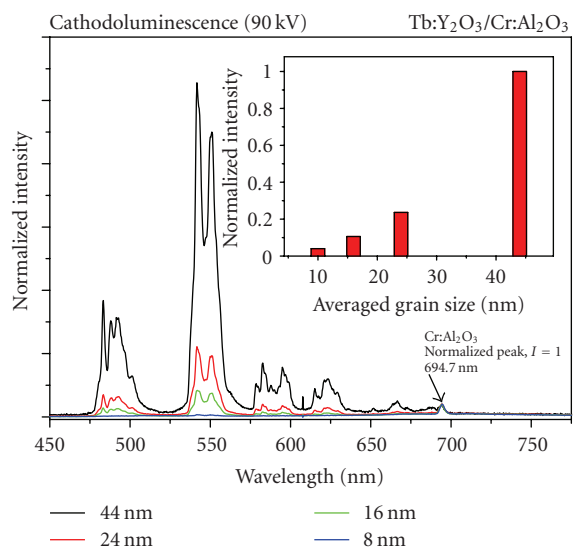


FIGURE 7: A comparison of cathodoluminescence (90 kV) of Tb:Y₂O₃/Cr:Al₂O₃ blend with different sizes of Tb:Y₂O₃ nanocrystallites.

3. RESULTS AND DISCUSSION

3.1. Structure

The XRD patterns of Tb:Y₂O₃ and Eu:Y₂O₃ nanocrystalline powders shown in Figures 1 and 2 were measured for the samples sintered at different temperatures. Following the Scherrer formula, we have determined the average grain sizes varying since 9 nm for 500°C to 46 nm for 900°C in the case of Eu:Y₂O₃ (see Figure 1(a)), and since 10 nm to 50 nm in the case of Tb:Y₂O₃ (see Figure 1(b)).

3.2. Cathodoluminescence

All the samples under investigation have demonstrated the low-voltage cathodoluminescent properties. The intensity of

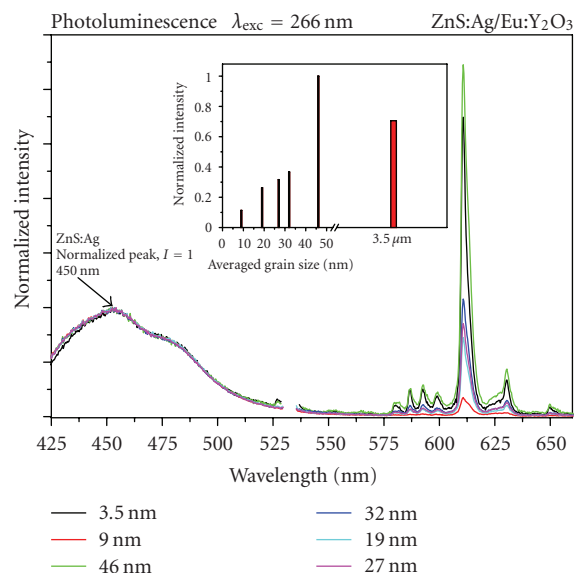


FIGURE 8: A comparison of photoluminescence ($\lambda_{\text{exc}} = 266$ nm) of Eu:Y₂O₃/ZnS:Ag blend with different sizes of Eu:Y₂O₃ nanocrystalites. A comparison of Eu:Y₂O₃ intensity has been presented in the middle of the figure.

emitted light has increased with increasing applied voltage (see Figure 5). The mixed Tb:YAG (green) and Eu:YAG (red) powders have emitted the yellowish-white light (see Figure 4). The spectra of nanophosphors were grouped and depicted regarding colors to compare the spectra (see Figures 2 and 3).

3.3. Time stability

The time stability measurements on nanophosphors subjected to cathodoluminescence process have been carried out for the Ce:YAG nanophosphor. It was observed that after first switch on light, the cathodoluminescence intensity stabilizes after 50 minutes; the time stability of emitted light measured in the next 50-minute period was satisfying (see Figure 6). Following our earlier studies, we could suggest that this delay is connected with time needed for cold cathode activation. Some residual contaminations should be removed from CNT-ITO cathode surface.

3.4. CIE chromatic coordinates

The CIE chromatic coordinates were analyzed on example of red light emitting phosphors. It is commonly known that the coordinates and luminescent spectra depend strongly upon doped ion and host [18]. Both Ce³⁺ and Eu³⁺ could be very good examples as an experimental confirmation of this thesis. What is shown in this work analysis is focused on Eu³⁺ ion. The features of Pr³⁺ doped CaTiO₃ has been also presented and compared to Eu:SnO₂, Eu:Y₂O₃, Eu:Y₂O₂S (see Figure 9). The emitted color transforms from deep bloody red for Pr:CaTiO₃ by fire red for Eu:Y₂O₂S and Eu:Y₂O₃ to orange-red for Eu:SnO₂. However, all examined phosphors are able to use as red phosphors in FEDs and as component

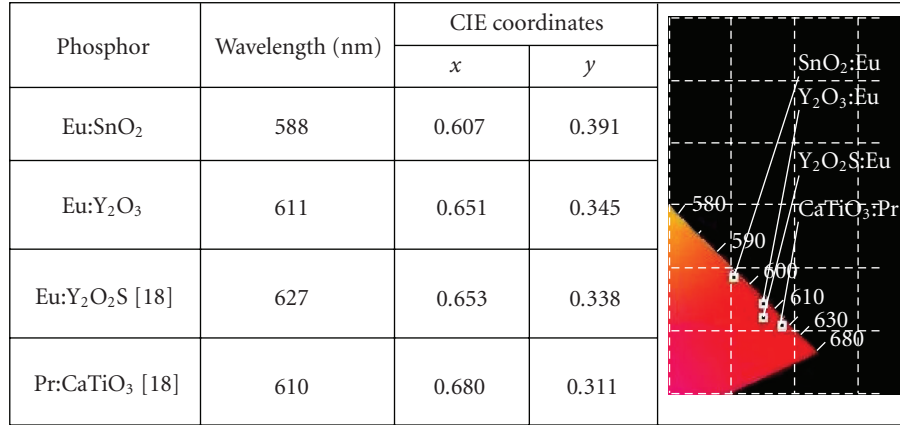


FIGURE 9: The comparison of CIE coordinate of different red phosphors.

of white light emitting phosphor for light source. Moreover, the Eu:SnO₂/Pr:CaTiO₃ and Eu:Y₂O₃/SnO₂ may be mechanically mixed with different mass ratios in order to tune the CIE coordinates of phosphor.

3.5. Size effects

The dependences of Cr:Al₂O₃/Tb:Y₂O₃ cathodoluminescence and ZnS:Ag/Eu:Y₂O₃ photoluminescence on the grain size are shown in Figures 7 and 8. The spectra were compared and normalized to the peak connected with the Cr:Al₂O₃ phosphorescence for cathodoluminescence measurements and to the peak connected with the ZnS:Ag luminescence in the case of photoluminescence. It is seen that in the nanophosphors, the light intensity increases with increasing average grain size. Moreover, a comparison of the photoluminescence spectra of micro- and nanograin sizes of ZnS:Ag/Eu:Y₂O₃ blend has demonstrated that the intensity of 3.5 μ m grain size powder of Eu:Y₂O₃ (Phosphor Technology) was about 70% of intensity of 46 nm grain size of europium doped yttria nanocrystallites synthesized by modified Pechini method [25, 27, 40, 51, 52]. An enhancement of the emission with grain size which is related to sintering temperature could be rationalized by a concept of the effective refractive index. It is well known that the radiative processes in nanocrystals of oxides doped with rare-earth ions strongly depend on the size, shape, and refractive indices of their surroundings [60–62]. The influence of the grain sizes on the luminescence lifetimes of Nd³⁺ doped YAG particles was earlier observed by Christensen et al. [60]. The emission intensity I is directly related with the Einstein coefficient for spontaneous emission A according to $I = h\nu AN$, where N is the population of centers emitting the energy $h\nu$. The Einstein coefficient A is simply related to the effective refractive index n_{eff} by the relation $A \propto \chi S$, where $\chi = (n_{\text{eff}}(n_{\text{eff}}^2 + 2)^2)/9$ and S is the dipole strength. Following the model of Meltzer [61], we may assume that the effective refractive index $n_{\text{eff}}(X) = x n_{\text{grain}} + (1 - x) n_{\text{surr}}$ is related to the filling factor x which shows the fraction of the sample

volume occupied by nanocrystals (n_{grain} is the refractive index of the material of the grain, n_{surr} is the refractive index of the medium surrounding the nanograins). Since for the same mass of powders with the same concentration of luminescent ions the filling factor is less for nanosize grains compared to microsize grains, the effective refractive index will be bigger for nanophosphors.

4. CONCLUSIONS

The results of present work clearly indicate that the nanocrystalline RE doped phosphors may effectively replace the microsize phosphors in FED devices. It is obvious that the layer made of nanocrystallites can be much thinner compared to the layer made of microsize phosphor grains. That has an impact both on total resistivity of the layer and amount of residual gasses bounded between the grains. Both these factors influence the efficiency of the light source, long time stability of the intensity of emitted light, and device lifetime. The light intensity of examined phosphors was stable during measurements. The emitted light intensity had increased with increasing applied voltage. A comparison of photo- and cathodoluminescent intensities of the layers and powders allows concluding that in the nanorange, the emitted light efficiency increases with increasing grain size. The second observation was that Eu:Y₂O₃ nanopowder (900°C, 46 nm) was more efficient than commercially available micrograin size phosphor (3.5 μ m). The CIE chromatic coordinates did not change with phosphor grain sizes.

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