

# Multicolor Depth-Resolved Cathodoluminescence from Eu-Doped SiOC Thin Films

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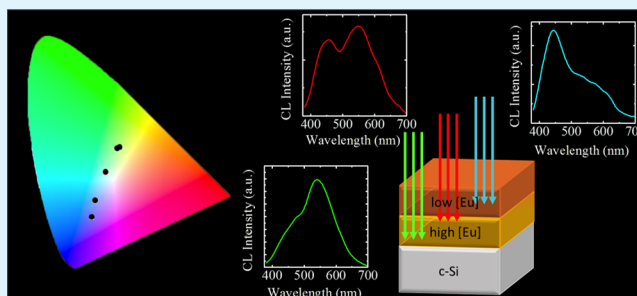
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**ABSTRACT:** A very bright room-temperature cathodoluminescence (CL) signal, tunable in the visible range by changing the Eu<sup>2+</sup> concentration, has been observed in Eu-doped SiOC films. Depth-resolved CL measurements demonstrate that a bilayer consisting of two SiOC films containing different Eu concentrations allows the continuous tuning of the Eu<sup>2+</sup> emission from blue to green by changing the energy of the exciting electrons. Furthermore, the proper control at the nanoscale of the electron penetration depth allows to obtain a high-quality white light emission. The compatibility of SiOC films with Si technology opens the way to promising applications of Eu-based materials in lighting and display technologies.

**KEYWORDS:** cathodoluminescence, rare earth ions, lighting, field-emission display, phosphors, photonics



Among the different approaches used for producing flat panel displays, an important role is played by field-emission displays (FEDs),<sup>1,2</sup> which are based on the irradiation of phosphors by electrons coming from a field emitter. The same working principle also operates in field-emission lamps (FELs), which attract considerable interest as mercury-free, efficient light sources.<sup>3</sup> Although it is true that in FED and FEL technologies many efforts are currently devoted to the development and optimization of the electron emitters, there are still very important issues related with phosphor properties, including the synthesis of thin-film phosphors exhibiting a full visible luminescence spectrum<sup>4</sup> and the matching of different luminescent layers for development of new principles of color variation.<sup>5</sup> Most of the phosphors used in FEDs are rare earth (RE)-doped oxides or sulfides; among all the available REs, a particular interest is devoted to Eu, owing to its characteristic to be stable both in its divalent and trivalent oxidation state. The very intense and tunable broadband emission produced by electric dipole-allowed  $4f^65d \rightarrow 4f^7$  transitions of Eu<sup>2+</sup> ions is generally more suitable for light source fabrication than the red emission coming from the dipole-forbidden intra  $4f$ -shell transitions of Eu<sup>3+</sup> ions, whose applications are limited by the relatively low efficiency.<sup>6</sup> Eu-doped materials may find interesting applications also in photonics, specifically for the realization of efficient, CMOS-compatible Si-based light sources operating at room temperature;<sup>7</sup> in particular, it has been recently demonstrated that Eu-doped SiOC films may play an outstanding role in this field. Indeed SiOC exhibits a remarkably higher Eu solid solubility than SiO<sub>2</sub>,<sup>8–10</sup> with the additional unique feature of favoring the formation of Eu<sup>2+</sup> ions instead of Eu<sup>3+</sup> ions, owing to the occurrence of a redox

reaction involving C atoms;<sup>8</sup> finally the system allows also to control the wavelength of the Eu<sup>2+</sup> photoluminescence (PL) signal from blue to red by changing the Eu concentration.<sup>11–14</sup>

In this work it will be shown that Eu-doped SiOC films exhibit a strong room temperature cathodoluminescence (CL) signal which can be tuned by changing the Eu concentration. The possibility to obtain an even wider color variation, spanning almost the entire visible range and, noteworthy, including also a high quality white emission, has been demonstrated by depth-resolved CL measurements, performed on a properly designed Eu-doped SiOC bilayer by changing the energy of the exciting electrons. For a deeper understanding of its potentialities, depth-resolved CL emission has been studied as a function of the electron beam current and compared with PL emission. The perspectives of Eu-doped SiOC films and multilayers in FED and lighting devices will also be addressed, highlighting the additional opportunities given by the compatibility of this material with Si CMOS technology.

An Eu-doped SiOC bilayer, consisting of two SiOC films having the same thickness (120 nm), but different Eu contents ( $3.0 \times 10^{21}$  Eu/cm<sup>3</sup> in the bottom layer,  $2.0 \times 10^{20}$  Eu/cm<sup>3</sup> in the top layer) was deposited by UHV RF magnetron cosputtering on a Si substrate. More details about the synthesis can be found in (14). As a reference, two single SiOC layers (with the same two Eu concentrations present in the bilayer) were also grown. All layers have a fixed C concentration of about 5 at. %. Samples were annealed at 750 °C for 1 h in N<sub>2</sub>

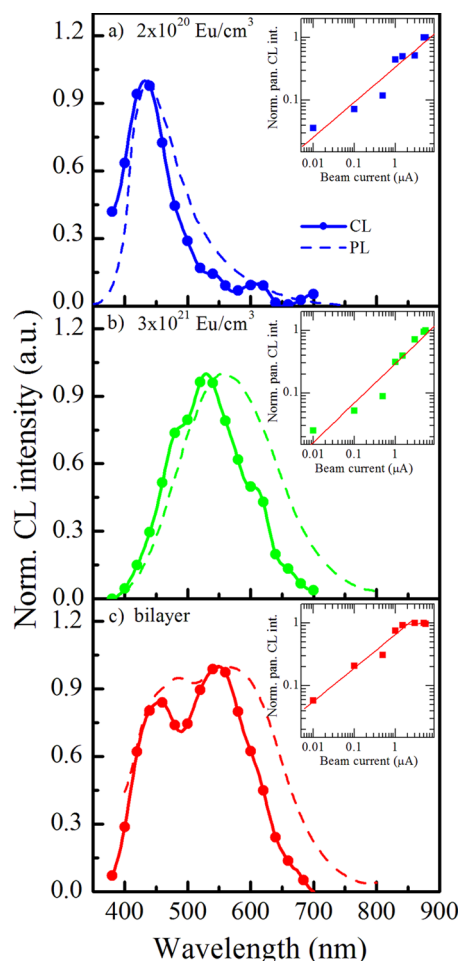
Received: June 16, 2015

Accepted: August 10, 2015

Published: August 10, 2015

ambient in order to maximize the room temperature luminescence efficiency of the Eu-doped layers.<sup>8,14</sup> The composition of the films was determined by Rutherford backscattering spectrometry (RBS) of 2 MeV He<sup>+</sup> ions. PL measurements were performed at room temperature by pumping with the 325 nm line of a He–Cd laser. The pump power was about 1.5 mW and the laser beam was chopped through an acousto-optic modulator at a frequency of 55 Hz. A commercial system, fitted onto a scanning electron microscope, was used for CL measurements. The system is equipped with a grating and a multialkali photomultiplier sensitive in the range 350–830 nm. Depth-resolved CL study was carried out varying the electron beam energy from 2.25 to 6 keV; the electron beam current was also varied in order to keep constant the injected power density (i.e., the injected power divided by the electron interaction volume defined by Monte Carlo simulations).<sup>15,16</sup>

To understand the perspectives of SiOC:Eu films in field-emission displays and lighting technologies, a study of their CL properties has been performed. In Figure 1a, b the CL spectra of SiOC layers doped with two different Eu concentrations (2.0



**Figure 1.** Room-temperature normalized CL (continuous lines and markers) and PL (dashed lines) spectra for Eu-doped SiOC single layers containing (a)  $2.0 \times 10^{20}$  Eu/cm<sup>3</sup> and (b)  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup> and for (c) Eu-doped SiOC bilayer containing  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup> in the bottom layer and  $2.0 \times 10^{20}$  Eu/cm<sup>3</sup> in the top layer. The insets show the dependence of the panchromatic CL intensity on electron beam current for each sample.

$\times 10^{20}$  and  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup>, respectively) are shown as continuous line and markers. The spectra, obtained by using a 3 keV electron beam as excitation source, mainly consist of a broad band; maxima are remarkably shifted one each other and are found respectively at about 440 and 530 nm and a longer emission wavelength corresponds to a higher Eu concentration.<sup>14</sup> Indeed, differently from the typical intra 4f transitions of trivalent RE ions, the emission from Eu<sup>2+</sup> ions is the result of a transition between a 4f<sup>6</sup>5d excited state and a 4f<sup>7</sup> ground state. Since 5d electrons are not efficiently shielded by 5s and 5p, the intensity and position of the emission peaks due to these transitions are strongly dependent on the host matrix composition and RE concentration; in particular, according to the crystal field theory, dopant incorporation into a host and the increase of its concentration can induce structural alterations, such as a change in bond lengths, which usually cause a redshift in the emission peak.<sup>11–14</sup>

In Figure 1a, b CL emissions are compared with the PL ones, obtained by using UV photons as excitation source, shown as dashed lines. Figure 1a highlights that the CL spectrum is basically identical to the PL one, except for the long wavelength region where a peak at around 615 nm, corresponding to the <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>2</sub> transition typical of Eu<sup>3+</sup> ions,<sup>17,18</sup> appears. The same phenomenon is also observable in Figure 1b. This difference between CL and PL spectra can be explained by taking into account an ionization process of RE<sup>2+</sup> ions induced by the high energy of the electron beam (2–6 keV) during the CL analysis,<sup>19–21</sup> analogously to the process of photoionization commonly observed in RE ions. According to this mechanism, previously observed for both Sm<sup>2+</sup> and Eu<sup>2+</sup> ions,<sup>19,20</sup> under electron beam irradiation the oxidation of Eu<sup>2+</sup> to Eu<sup>3+</sup> may occur; note also that this phenomenon can lead to a shift of the 5d shell of Eu<sup>2+</sup>, which is strongly dependent on the ion environment, explaining also the slightly different peak position between the CL and PL spectra in Figure 1b. In the case of PL, ionization cannot occur because of the remarkably lower energy (3.8 eV) of exciting photons.

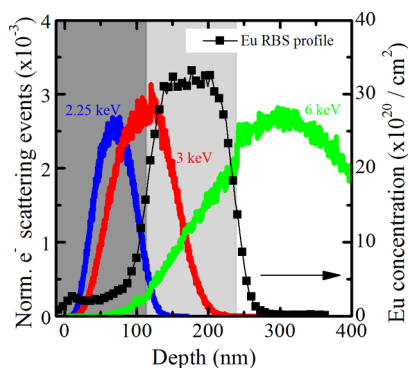
To test the potentialities of Eu-doped SiOC as an efficient field emission source of white light, we have investigated the behavior under electron irradiation of a bilayer composed by the two SiOC:Eu layers discussed in Figures 1a, b. Figure 1c shows that the CL spectrum of the bilayer consists of a very wide band, covering almost the entire visible range, exhibiting two maxima. From the comparison with the CL spectra of the two reference layers in Figures 1a, b, it is straightforward to assign the component exhibiting the maximum at shorter wavelength to the layer with  $2.0 \times 10^{20}$  Eu/cm<sup>3</sup> and the other one to the layer with  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup>. Again the PL and the CL emission spectra are slightly different between each other and also in this case in the CL spectrum it can be noticed a shoulder at around 615 nm due to the presence of Eu<sup>3+</sup>.

The insets of Figures 1a–c show the dependence of the panchromatic CL intensity (integration range 300–900 nm) on the electron beam current in the range between 10 nA and 5  $\mu$ A for the two single layers and for the bilayer, respectively. During CL measurements, the voltage has been kept constant at 3 kV. The insets show that the panchromatic CL intensity of the two single layers increases with the current, while in the case of the bilayer the initial behavior approaches to saturation above 1  $\mu$ A. The dependence of CL intensity (*I*) on electron beam current density (*j*) can be described by a power law  $I \propto j^m$ .<sup>22</sup> The *m* values given by the linear fits to the data plotted in a log–log scale in the three insets, shown as continuous lines,

are very similar (by excluding from the fit procedure the saturation region of the bilayer) and lie in the 0.50–0.65 range. Values of the exponent  $m$  smaller than one indicate the presence of both radiative and nonradiative centers in the films;<sup>5,23</sup> furthermore, the similar values of  $m$  found for all the three samples confirm that, as expected, the same balance between radiative and nonradiative processes is reached in films which only differ for the dopant concentration. Consequently, the saturation behavior at high beam currents exhibited by the bilayer is not due to an intrinsic property of the material, but only to a difference of thickness; indeed, saturation in CL intensity may result from electron-induced charging due to the insulating environment,<sup>24</sup> because of the larger distance from the Si substrate of electrons into the bilayer with respect to those into the single layers, for the same current and accelerating voltage.

The data shown in Figure 1 demonstrate the capability of Eu-doped SiOC films to act as a visible light source in a field emission device; the wavelength of the emitted light can be tuned by playing on the Eu concentration of a single layer, or by properly combining layers emitting at different wavelength. However, the possibility of changing the electron penetration depth by changing their energy opens new perspectives with respect to photon excitation of the same material; indeed, it should be possible to obtain, from the same SiOC multilayer, emission at different wavelengths by simply controlling the region of the sample in which most of the scattering events occurs.

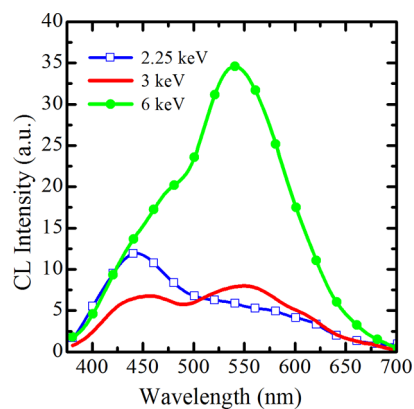
To demonstrate its capability to operate as a variable color light source, we have investigated the CL properties of the SiOC:Eu bilayer for different electron beam energies. The color change is achieved by changing the electron penetration depth through the variation of the electron beam energy. To illustrate the depth-sensitivity of the electron-material interactions, Figure 2 shows the distribution of electron scattering events,



**Figure 2.** Simulated scattering events versus depth for electrons penetrating in the Eu-doped SiOC bilayer with energies of 2.25, 3, and 6 keV. The Eu concentration profile obtained by RBS is also shown as black line and solid squares. The dark gray region corresponds to the top Eu-doped SiOC layer with  $2.0 \times 10^{20}$  Eu/cm<sup>3</sup>, whereas the light gray region corresponds to the bottom Eu-doped SiOC layer with  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup> and the white one to the c-Si substrate.

obtained from Monte Carlo simulations,<sup>25</sup> for electron beam energies of 2.25, 3, and 6 keV; the density of SiOC:Eu thin films used for the simulations has been estimated by the RBS measurements. The Eu concentration depth profile obtained by RBS is also shown as black line and solid squares. The scattering distributions correspond to the sample regions where

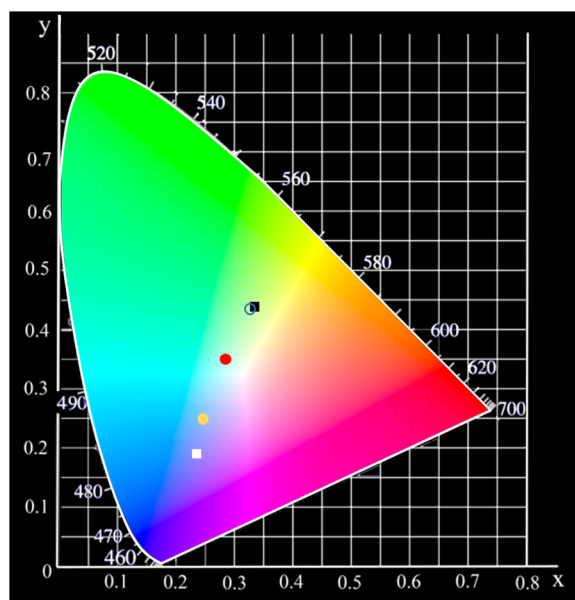
most of energy deposition, and therefore of CL signal generation, occurs; however, due to secondary scattering events, the true interaction volumes are larger and have diffuse boundaries. The figure illustrates the ability to probe continuously from the upper layer (dark gray region), to the bottom one (light gray region), by varying the accelerating voltage between 2.25 and 6 kV. In particular, for accelerating voltages up to 2.25 kV (blue line), the electron beam is almost totally stopped into the top SiOC:Eu layer, which has a lower Eu concentration. By increasing the electron beam energy to 6 keV (green line), electrons penetrate more deeply into the sample, generating scattering events falling mostly in the bottom layer, having the highest Eu concentration. At the intermediate acceleration voltage of 3 kV (red line) the beam penetrates through the film up to 200 nm with a maximum in the number of scattering events at 120 nm. In these conditions, the volume of the electron scattering events involves both layers; as a consequence, the CL emission spectrum at 3 keV, shown in Figure 3 as a continuous red line, is the result of a



**Figure 3.** Room-temperature CL emission spectra of the Eu-doped SiOC bilayer excited with electron beam energies of 2.25, 3, and 6 keV.

combination of the emission from both layers, as already discussed, and it consists of a broad band with two maxima, at 440 and 540 nm, which roughly have the same intensity. Figure 3 shows also the CL emission obtained for electron beam energies of 2.25 and 6 keV; CL emission at 2.25 keV (blue line and squares) shows a spectrum with a marked peak at 440 nm, which closely resembles the emission from the single layer with lower Eu concentration (Figure 1a), while by increasing the electron beam energy up to 6 keV (green line and circles) the emission appears peaked at 540 nm, again resembling the spectrum of the layer with higher Eu concentration (Figure 1b). Note that spectra taken at 2.25 and 6 keV have also a shoulder at higher and lower wavelength, respectively, mainly due to the contribution of secondary scattering events, which are not taken into account in the simulations of Figure 2, leading to a broadening of the real interaction volume of the electrons into the material.

For possible practical applications in display and lighting technologies, it is appropriate to analyze the emission spectra of the SiOC:Eu bilayer in terms of the Commission International de l'Éclairage (CIE) chromaticity coordinates. Figure 4 shows the chromaticity diagram in which the points corresponding to the CL spectra of the bilayer, for all the investigated electron beam energies, are reported as circles. We can observe that the emission range spans from blue to green-yellow by increasing the electron beam energy. For an energy of 2.25 keV (yellow



**Figure 4.** CIE chromaticity coordinates, shown as circles, for the Eu-doped SiOC bilayer obtained by analyzing the CL emission detected for electron beam energies of 2.25 (yellow closed circle), 3 (red closed circle), and 6 (blue open circle) keV. The chromaticity coordinates of the two Eu-doped SiOC with  $2.0 \times 10^{20}$  and  $3.0 \times 10^{21}$  Eu/cm<sup>3</sup> reference layers, excited with a 3 keV electron beam energy, are also shown as white and black squares, respectively.

closed circle), a blue emission with coordinates (0.25, 0.25) is achieved, whereas at 6 keV (blue open circle) a green-yellow emission with coordinates (0.31, 0.43) is obtained. A white emission is obtained at 3 keV (red closed circle); its coordinates (0.30, 0.36) are very close to those relative to an ideal white emission, which are (0.33, 0.33). As expected, in the diagram, the coordinates relative to the bilayer lie on the straight line connecting the points corresponding to the reference layers analyzed at 3 keV, shown as squares at the coordinates (0.24, 0.20), for the blue emission of the reference layer with the low Eu content (white square), whereas the layer with the high Eu content (black square) is found at (0.31, 0.42).

From the CIE coordinates, it is also possible to calculate the correlated color temperature (CCT) of the emission; for the bilayer emission at 3 keV, the calculated CCT is 6086 K. CCT is typically used to classify the light emitted by commercial LEDs and lamps and a value around 6000 K is typical of a daylight source. It is also possible to evaluate the color rendering index (CRI), which is a measure of the capability of a light source to correctly render all colors, because it represents the difference between the actual emission and that one theoretically coming from an ideal emitter at a given temperature (6086 K in the present case). For the Eu-doped SiOC bilayer excited with an electron beam energy of 3 keV we have calculated a CRI value of 70/100, which mirrors the broadband nature of the Eu<sup>2+</sup> emission. The above CCT and CRI values are very close to the ones of commercial fluorescent lamps, suggesting that this system may be suitable for practical lighting applications.

In conclusion, we have demonstrated that Eu-doped SiOC thin films can be promising candidates for FED applications; furthermore, the evidence that, through a proper tuning of the energy of the exciting electron beam, an Eu-doped SiOC bilayer

is a light emitter able to continuously tune its emission from blue to green and also to emit a high-quality white light, opens the way also for applications of this material in lighting, as the active layer in field-emission lamps. Although the variation of the emitted light from a single multilayered phosphor by controlling at the nanoscale the penetration of the exciting electrons has been previously reported,<sup>5</sup> in our approach this result is achieved by putting together two very similar layer, differing only for the dopant concentration, so providing the additional advantage to avoid any chemical or structural incompatibility between the bilayer components. Furthermore, the compatibility of the SiOC matrix with Si CMOS technology makes Eu-doped SiOC layers and multilayers also a promising candidate for bridging the gap between FED and CMOS technologies.

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### Notes

The authors declare no competing financial interest.

## ■ ACKNOWLEDGMENTS

The authors thank F. Priolo, G. Salviati, and S. Boninelli for fruitful discussions.

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