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$SrGa_2S_4$: RE phosphors for full colour electroluminescent displays

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Abstract

Ternary thiogallate sulphides doped with rare-earth ions fulfill the electrical and optical requirements for thin film electroluminescent phosphors. These materials represent a breakthrough in the achievement of full colour EL displays. $SrGa_2S_4$ provides a deep blue emission when doped with Ce^{3+} and a saturated green with Eu^{2+}. Some luminescent properties of Ce^{3+} and Eu^{2+} in $SrGa_2S_4$ compounds are reviewed and the various techniques used to prepare thin films of this ternary compound are presented. The behaviour and performances of EL devices are discussed. © 1998 Elsevier Science S.A.

Keywords: Electroluminescence; Thin films; Ternary compounds; Rare earth; Phosphors

1. Introduction

Thin Film Electroluminescent (TFEL) displays represent one of the most attractive techniques for emissive flat panels [1]. The most efficient host matrices for EL are the II–VI sulphides or selenides of Zn, Ca and Sr, the best phosphor being the well known yellow ZnS:Mn phosphor. By filtering this large band emission, marketing of colour EL displays, green–yellow–red, has been achieved. Recently the use of the $Zn_{0.9}Mg_{0.1}S$:Mn phosphor has improved the colour range [2].

The dominant approach to succeed in full colour TFEL displays is 'colour by white' based on the stack of SrS:Ce/ZnS:Mn phosphors (Fig. 1a) [3,4]. Ce^{3+} in SrS provides a blue-green emission; in recent years, intensive efforts on the preparation of the emitting layer have been successful in increasing the brightness and the efficiency of this material [5]. Prototypes have been presented and these colour displays are expected in the market in the near future. But the colour gamut defined by the three colour filters is not adequate for full colour applications, specially for the blue and the brightness still needs to be improved.

The second approach is the patterned phosphor in a dual substrate structure (Fig. 1b) [6]. The development of efficient EL phosphors for the primary red, green and blue colours has been the challenge of TFEL research during the last ten years. The standard ZnS:Mn yellow phosphor and a red filter provides an efficient red. For the green, the ZnS:Tb phosphor also gives good efficiency. The problem of the blue was not well solved and the need for new host matrices was urgent and critical. Recently, in 1993, the fabrication of TFEL devices based on alkaline earth thiogallate phosphors activated with Ce^{3+} and Eu^{2+} have made a breakthrough in phosphors for full colour TFEL panels [7,8].

In this review, we discuss the requirements for TFEL phosphors which explain the choice of the ternary compounds; then we present some characteristics of these phosphors, the synthesis of these films by different techniques and the electro-optical behaviour of $SrGa_2S_4$ EL devices.

2. The choice of the M^{IIA} M_2^{III} $(S,Se)_4$ ternary compounds

The choice of a new host-matrix has to be guided by the particular requirements for TFEL phosphors.

electrical: an efficient charge carrier acceleration to optical energies under high electric fields to excite luminescent centers.
optical: an efficient phosphor in the visible range.

2.1. Electrical requirements

In order to determine the energy distribution of the carriers in ZnS based ACTFEL devices, models for high

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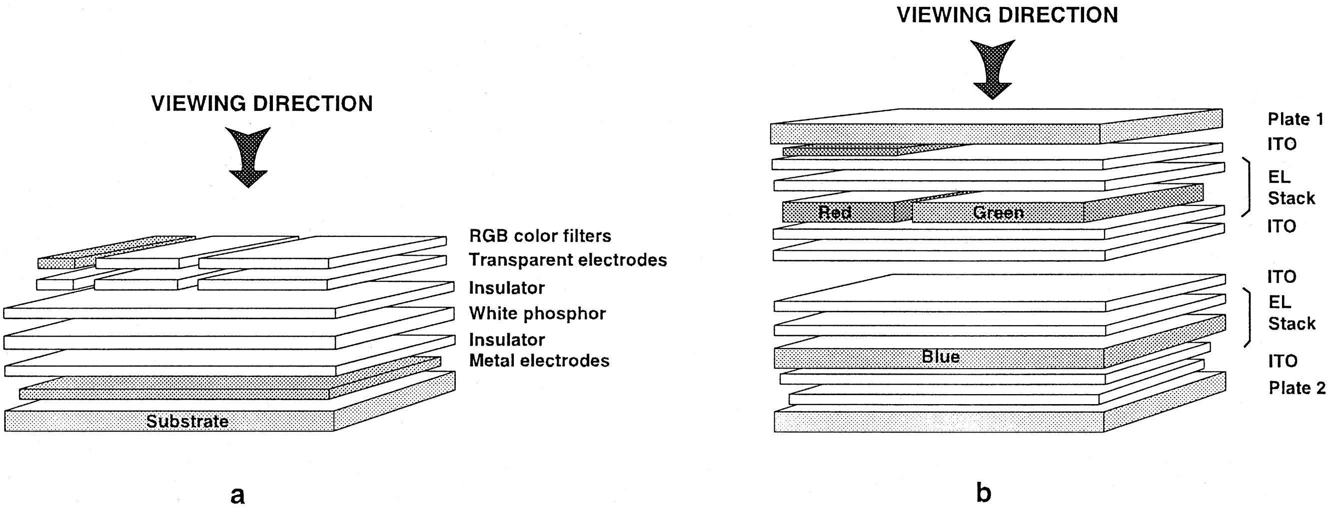

Fig. 1. Two colour thin-film EL structures [1]. (a) Patterned phosphor scheme in a dual-substrate structure. (b) Colour-by-white scheme in an inverted structure.

field charge transfer have been developed [9,10]. Above an electric field of the order of 1 MV cm^{-1}, electron–phonon interaction is the predominant scattering mechanism. Compared to oxygen dominated compounds, sulphides exhibit rather low LO phonon energies (Table 1) [11–14], and seem therefore to provide good properties for TFEL. This has been confirmed in the case of binary sulphides. However, the band structure of the material also plays an important role in the acceleration process because it determines the density of final states for electron diffusion process and therefore the diffusion rate.

In fact, no strict a priori criterion exists to select an adequate material for EL host matrix, except the fact that sulphide compounds, in contrast to oxygen ones, present low LO phonon energies.

2.2. Optical requirements

The semiconductor must have a large band gap, more than 3 eV, in order to be transparent for an emission in the visible range, condition necessary also for the semiconductor to support a high electric field over 10^6 V cm^{-1}.

As to the dopant, only emissions belonging to inner shell transitions are efficient under high electric fields, and one will consider the Mn^{2+}, Ln^{2+} and Ln^{3+} ions. The emissions provided by these ions must have good chromaticity coordinates corresponding to one of the three R–G–B primary colours for visualisation. The emissions of Ce^{3+} and Eu^{2+} concern allowed d→f transitions. The energy of these emissions are strongly influenced by the host-matrix due to the external 5d orbitals. Therefore, the possibility of matching the emissions in binary sulphides host matrices is limited and unsatisfactory, and it was logical to look at ternary sulphide compounds. By considering all these aspects, the ternary sulphide and selenide compounds $M^{IIA}M_2^{III}$ (S,Se)$_4$, where IIA=Mg, Ca, Sr, Ba and III_2=Al, Ga, In, Y have been evaluated as good candidates for TFEL phosphors.

3. Main characteristics of M^{IIA} M_2^{III} (S,Se)$_4$ phosphors

Luminescent properties of M^{IIA} M_2^{III} (S,Se)$_4$ compounds, doped with different dopants, especially Eu^{2+} and Ce^{3+} have been studied since the 70's [15–21]. Although some of them are known as efficient phosphors, few data have been published. Some of the ternary sulphides were studied in DC powder panels [22].

The Ce^{3+} emission presents two overlapping bands corresponding to transitions from the 5d state to the spin orbit split states $^2F_{5/2}$ and $^2F_{7/2}$ of the $4f^1$ configuration separated by about 2000 cm^{-1}. The Eu^{2+} emission con-

Table 1
LO phonon energies of some materials

Material	LO phonon energies (meV)	Material	LO phonon energies (meV)
SrS	37 [11]	$PbGa_2S_4$	48, 37 [14]
CaS	47 [12]	La_2O_2S	57 [12]
ZnS	44 [12]	Y_2O_3	68 [12]
ZnO	72 [13]	$Y_3Al_5O_{12}$	105 [12]
ZnSe	31 [13]	Zn_2SiO_4	130 [12]

sists of a band belonging to the transition from the lower $4f^6 5d^1$ state to the $4f^7$ ($^8S_{7/2}$) ground state. As both emissions are strongly dependant on the environment of these two ions [23], the energy of the emission will be determined by the choice of the M^{II} and M^{III} elements. For example, the energy of the lower Eu^{2+}5d level in these compounds decreases with increasing size of M^{III} (Al, Ga, In) and with decreasing size of the M^{II} cation (Ba, Sr, Ca). The 5d→4f emission band peaks over the 467–516 nm range for thioaluminates, the 495–560 nm range for thiogallates and in the deep red for thioindiates [18]. For the latter hosts, thermal quenching occurs well below room temperature. In contrast, Eu^{2+} doped thioaluminates and thiogallates show high efficiency at 300 K, owing to the increase in the bandgap in the following order of the trivalent elements: In<Ga<Al [18,19]. Only a weak photoluminescence has been reported for homologous selenium compounds of thiogallates [19]. The Eu^{2+} emission in MY_2S_4 phases lies in the orange–red region; it is markedly affected by thermal quenching at 300 K [24].

Present data for Ce^{3+} are restricted to thiogallates. The main peak emission varies from 455 nm for M^{II}=Ba, Sr to 468 nm for M^{II}=Ca [15–18].

Unlike thioaluminates, thiogallates are not very sensitive to the action of air moisture and their stability decreases in the order Ba>Sr>Ca [18].

In conclusion, $SrGa_2S_4$ is weakly hygroscopic; doped with Eu^{2+}, it yields better green emission compared to the binary sulphides EL host matrices; when doped with Ce^{3+}, compensated with Na^+, it provides a deep blue which does not require any colour filter. These are the main reasons why Sr and also Ca thiogallates were selected to examine whether the ternary sulphide compounds fulfil the requirements for a TFEL host matrix.

The promising EL results obtained with $SrGa_2S_4$ thin films have given rise to new studies on powders in order to get a deeper knowledge of the luminescent properties of Ce^{3+} and Eu^{2+} ions in this material, and also to have reference data to evaluate the quality of the films. Detailed investigations of the photoluminescence and cathodoluminescence properties of Eu^{2+} and Ce^{3+}, Na^+ doped $SrGa_2S_4$ powders have been performed [25,26]. The main results for Ce^{3+} are:

a concentration of 4 mol % is a good compromise for the blue phosphor between the increase of the luminescence (optimal content 10%, Fig. 2) and a green shift of the spectra with Ce^{3+} concentration.

a low concentration quenching and no thermal quenching up to 350°C for 4 mol % concentration.

under cathodic excitation, the luminescence of intensity increases linearly with the current density up to 60 $\mu A\ cm^{-2}$ (Fig. 3).

All these characteristics makes Ce^{3+} and Eu^{2+} doped $SrGa_2S_4$ phosphor powders good candidates for use in high current density cathodic screens like field emission display (FED) or projection TV [27].

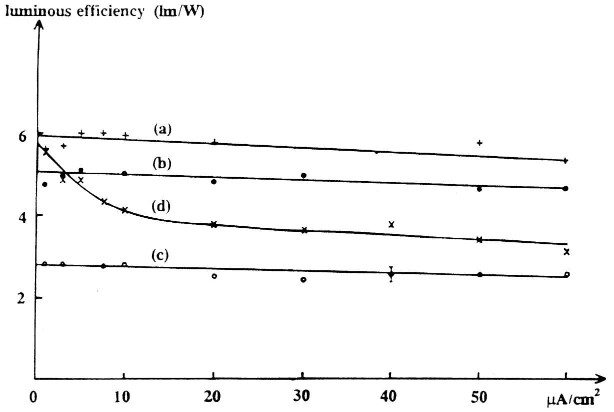

Fig. 3. Luminous efficiency of $Sr_{1-2x}Ce_xNa_xGa_2S_4$ phosphors and ZnS:Ag, Cl (Nichia 54TC) versus current density. Measurements were performed with an electron beam scanned at 50 Hz over a 2×2 cm raster and accelerated under 16 kV. (a) x=0.10, (b) x=0.06, (c) x=0.02, (d) ZnS:Ag, Cl [24].

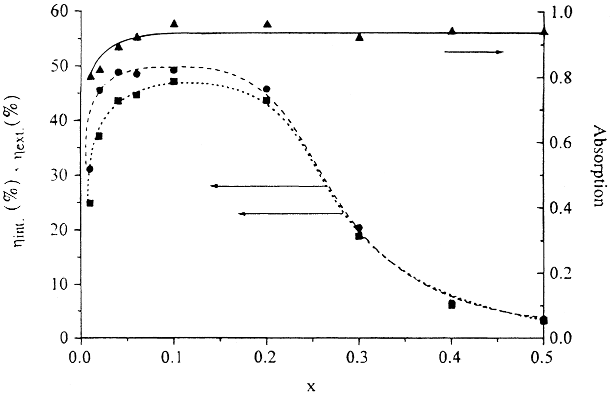

Fig. 2. Cerium concentration dependence of the internal (•) and external (■) photoluminescence quantum efficiency for the series $Sr_{1-2x}Ce_xNa_xGa_2S_4$ at 300 K (λ_{exc}=409 nm). Also included is the variation of the absorption A (▲) (derived from reflectance spectra) [24].

4. $SrGa_2S_4$ luminescent thin film synthesis

Ternary sulphides are chemically more stable than alkaline earth sulphides. II–VI and III–VI bonds are present in $SrGa_2S_4$, the binding energy of each one being significantly different. Hence, the stoichiometry of the ternary compounds will be a crucial challenge to succeed by controlling the supply of the three elements plus the dopant and eventually codopant elements. Vecht has recently reviewed the synthesis of thin films phosphors based on ternary systems and his conclusion was that this question “has been largely neglected” [28].

Sputtering was the first technique used to prepare $SrGa_2S_4$ thin films [7,8], but also other methods were carried out, such as molecular beam epitaxy (MBE), multisource vapour deposition (MSD) and MOCVD.

4.1. Sputtering

The sputter target can be prepared by sintering phosphor powder or at least by sintering a mixture of SrS, Ga_2S_3 and EuS or $Ce_2S_3^+$NaS powders. Reactive sputtering with 1–3 mol.% of H2S in an argon atmosphere was necessary as electron probe microanalysis (EPMA) has revealed a lack of sulphur for films prepared in pure argon atmosphere. The film composition was analyzed by EPMA in the case of $SrGa_2S_4$:Eu^{2+} [29]. A better composition of the film was obtained for a temperature of the substrate between 100–150°C, a H_2S content between 2–3%. Samples annealed below 550°C after deposition showed only an amorphous structure under XRD and not any luminescence. Increasing the annealing temperature up to 700°C over 2 h under vacuum, crystallization and luminescence intensity increase. The PL emission of these $SrGa_2S_4$:Eu^{2+} thin films show a broad band centered at around 531 nm. For a very short (0.6 ns) photonic excitation at 400 nm in the lower Eu^{2+} absorption band, the decay exhibits a small fast non-exponential part followed by an exponential with $\tau_0=490$ ns for more than two decades (Fig. 4). The normalized area under the decay curve, S_N, takes a value of 450 ns close to the value measured for powder samples (480 ns) [29]. In the case of RF sputtering from a Ce doped thiogallate phosphor target, a post-deposition rapid thermal anneal at temperatures above 650°C produced complete crystallization [8].

The very important annealing temperature leads to several problems: one needs substrates with a high strain point and the layers under the phosphor film, the first electrode and the first insulator have to support this annealing without damage.

4.2. Other techniques

Considering the other physical and chemical deposition techniques, we can expect that stoichiometric composition can be obtained at a low temperature by the accurate source flux control and the chemical reaction of the source materials. Different arguments are developed to use either the three elements Sr, Ga and S of the host matrix [30] or binary vapors [31].

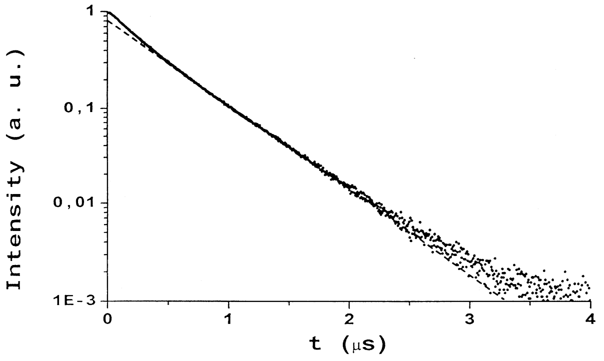

Fig. 4. The PL decay curve of $SrGa_2S_4$:Eu^{2+} thin film. $\lambda_{exc}=400$ nm, $\lambda_{anal}=530$ nm [27].

MSD technique has provided efficient EL $SrGa_2S_4$:Ce layers by coevaporation of the three elements, Sr, Ga and S of the host matrix, $CeCl_3$ as dopant source and LiF as a growth enhancing flux agent. Without a flux agent, Ga-rich phases and SrS phase segregation are present. By using Ga_2S_3 instead of Ga and S sources, the formation of the film follows the Ga_2S_3–SrS pseudo-binary line. The MSD technique has provided EL samples with a brightness of 78 cd m^{-2} at 1 kHz [30].

Deposition from binary vapors (DBV) has also provided stoichiometric blue emitting thin films in spite of the complete dissociation upon evaporation, of SrS and Ga_2S_3. A precise Ga_2S_3/SrS impingement flux ration has been determined for a substrate temperature of 460°C.

MBE technique, using Sr, Ga_2S_3 and $CeCl_3$ as source materials has provided EL samples exhibiting a brightness of 70 cd m^{-2} at 1 kHz for a substrate temperature of 540°C [32,33]

MOCVD has just been investigated [34] and more studies are necessary to evaluate the possibilities of this technique.

In conclusion, MBE and MSD techniques have provided promising results at lower temperature than sputtering. But it is crucial to obtain the unique $SrGa_2S_4$ phase not only for the radiative efficiency of the layer. The presence of other phases is detrimental to the acceleration process of the carriers as well as for the reliability of the EL device.

Thus far, the question of the most appropriate method to prepare thiogallate phosphor thin films remains open.

5. $SrGa_2S_4$ EL devices

To prepare a TFEL device, one has to choose the insulator material compatible with the phosphor host matrix material. For a classical MISIM structure, the insulator must have a higher dielectric constant than the host matrix and a high factor of merit, that is the product of the dielectric constant with the breakdown electric field [1]. Thiogallates have a high dielectric constant of the order of 15 [8] and thus the choice of the insulator materials is limited; $TiAlO_x$, $BaTa_2O_6$ or $SrTiO_3$ were selected due to their good reliability.

5.1. Spectra and colour coordinates

For the green emission, $SrGa_2S_4$:Eu^{2+} EL devices exhibit a band centered at 528 nm, the colour coordinates of this emission being $x=0.28$ and $y=0.69$. The brightness voltage curve is sharp and a maximum brightness of 10^6 cd m^{-2} was obtained at 500 Hz for a sinusoidal excitation voltage of 210 volts rms (Fig. 5 [29]). For this voltage, the saturation range is just approached. An increase of one order of magnitude more of the brightness is the require-

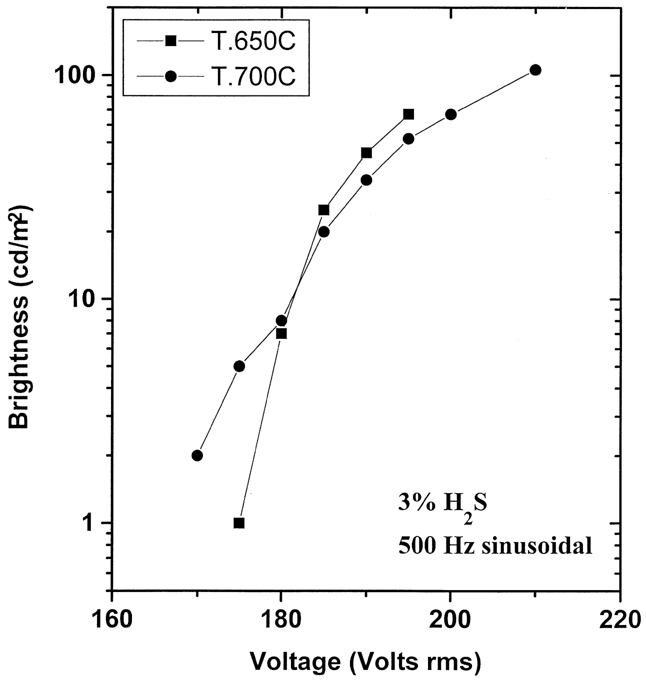

Fig. 5. $SrGa_2S_4:Eu^{2+}$ (2 mol%) EL device: brightness versus applied voltage [27].

ment for the use of this phosphor instead of ZnS:Tb as the green emitting phosphor for the full colour EL display.

The main efforts in EL studies have been focused on the blue phosphor and sputtering, MBE and MSD techniques have provided a similar brightness. The three thiogallates of Ba, Sr and Ca have been studied as TFEL phosphors (Fig. 6) [35]. The deeper blue, close to the standard CRT display, is furnished by the Sr compound. But the brightness is still too low for application and therefore a compromise has been made by choosing the Ca compound. The radiative efficiency is quite similar but the luminous efficiency is higher with Ca (Table 2) Fig. 7.

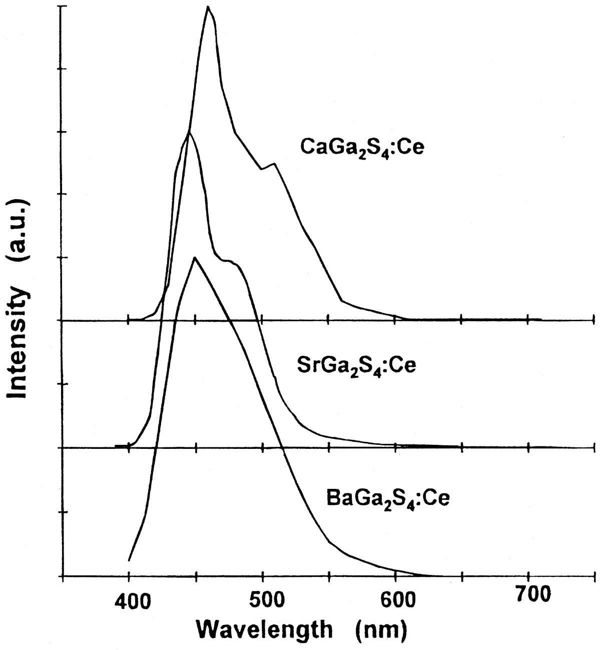

Fig. 6. EL spectra of Ce^{3+} activated thiogallate devices [33].

Recently a codoping of layers with O during the sputtering process has lead to an improvement in the brightness. It is assumed that the crystallinity was improved, some sulphur vacancy being suppressed by O^{2-} ions [36].

5.2. Excitation mechanism

The electric behaviour of thiogallates shows many of the characteristics known from ZnS based TFEL devices. By means of the bridge method, one can observe the time resolved charge transfer in the thiogallate layer; the light response is also recorded. This behaviour is independent of the dopant, Ce^{3+} or Eu^{2+} (Fig. 8) [29].

When the quality of the ternary compound is good enough, the charge transfer response is very fast, the decay constant of the decay being of the order of 1 μs, which corresponds to the time constant of the device. The light response follows the charge transfer due to the fast decay time of Eu^{2+} and Ce^{3+}. At the trailing edge of the pulse, when the voltage applied to the sample drops to zero, no second current peak or second emission is observed, even at high transferred charge. This result indicates that the investigated luminescent centers in $SrGa_2S_4$ are not ionized under the influence of the high electric field as with Eu^{2+} and Ce^{3+} in the alkaline earth sulphides [37,38]. In

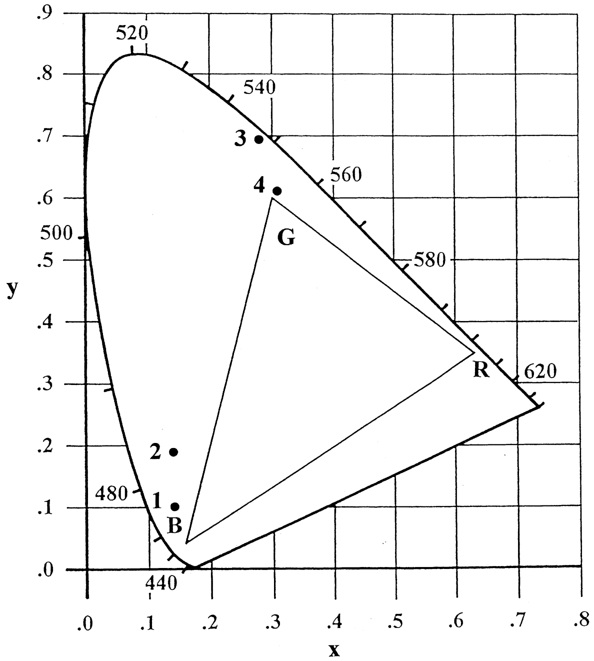

Fig. 7. CIE chromaticity diagram for EL phosphors. Blue phosphors: 1=$SrGa_2S_4:Ce^{3+}$; 2=$CaGa_2S_4:Ce^{3+}$. Green phosphors: 3=$SrGa_2S_4:Eu^{2+}$; 4=ZnS:Tb.

Table 2
Performances of ternary EL compounds[a]

Phosphor	Concentration at.%	CIE coordinates		Brightness L cd m^{-2} (60 Hz)	Luminous efficiency
		x	y		η (lm W^{-1})
$CaGa_2S_4$:Ce^{3+}	1	0.14	0.19	10	0.03
$SrGa_2S_4$: Ce^{3+}	1	0.14	0.10	5	0.02
$BaGa_2S_4$: Ce^{3+}	0.5	0.15	0.15		
$Ca_{0.5}Sr_{0.5}Ga_2S_4$:Ce^{3+}	1	0.14	0.13	4.3	
$SrGa_2S_4$:Eu^{2+}	2	0.28	0.69	106 (500 Hz)	

[a] [1]

this latter case, at the trailing edge, carriers move on the other direction due to the residual polarisation field and they recombine with the ionized centers which de excite to emit a photon. This different behaviour between binary and ternary sulphides could be explained by a different localization of the 5d level of Eu^{2+} or Ce^{3+} with respect to the conduction band.

6. Conclusion

$SrGa_2S_4$ thin films have been prepared by different methods. This material, like other thiogallates, allows the acceleration of charge carriers up to optical energies under high electric fields. It produces a deep blue emission when doped with Ce^{3+} and a saturated green with Eu^{2+}.

The excitation mechanism of these luminescent centers seems to be a one step direct impact excitation as in the ZnS:Mn, a result which allows one to forecast high excitation efficiency.

The brightness and the efficiency of the blue phosphor of MGa_2S_4:Ce have reached a level which has permitted the fabrication of true full colour EL panels [39].

The future of thiogallate EL devices requires an improvement of their performances. Technological progress on the properties of the substrate and the insulator layers can be expected. But the key is the quality of the phosphor layer, viz a vis stoichiometry, crystallography and non-disturbed symmetry of the sites of the dopants. These improvements will lead to an increase of in the radiative and excitation efficiencies.

Other ternary sulphide compounds could satisfy the EL requirements. In particular the barium thioalluminate $BaAl_2S_4$ presents a wider band gap than thiogallates, and doped with Eu^{2+} provides an intense blue emission with colour coordinates better than Ce^{3+} in $SrGa_2S_4$ [21]. The hygroscopicity of $BaAl_2S_4$ can be prevented by encapsulation between the two insulator layers. The first $BaAl_2S_4$:Eu^{2+} EL devices have been prepared by RF sputtering. But the crystallization of this ternary compound needs at least an annealing temperature of 800°C and several phases were present; therefore the brightness was very low [40].

In conclusion, the interest towards strontium thiogallate phosphors has been very strong during the last years to obtain the blue and the green primary colours for flat emissive displays, powder for FED and thin films for EL.

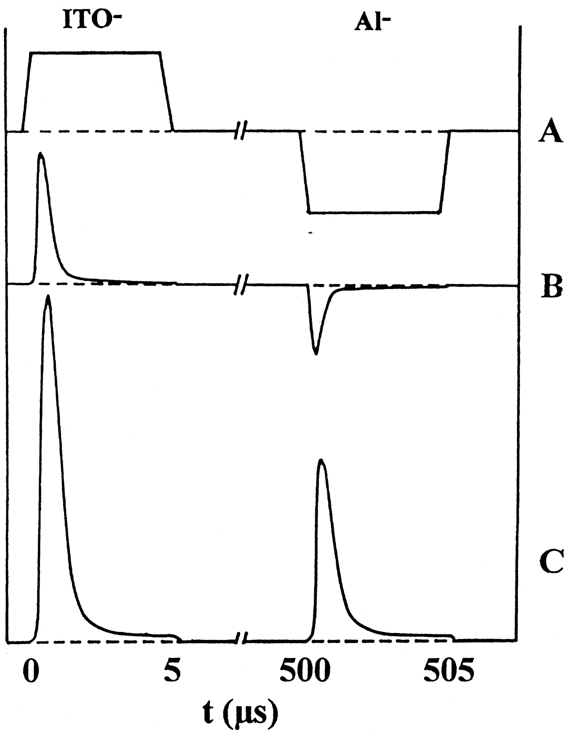

Fig. 8. Electric pulse (A), Current (B) and Optical response (C) in $SrGa_2S_4$:Eu^{2+} TFEL devices. Pulse width, 5 μs; rise and fall time, 0.5 μs [27].

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