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PHOSPHORS AND FLUORESCENT CONVERTERS IN LIGHT SOURCES WITH BLUE LED CRYSTALS

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ABSTRACT

The article presents a review of fluorescent material use: inorganic phosphors, organic polymeric and molecular phosphors, complex (coordination) compounds, quantum dots and frame metal-organic compounds as component materials of fluorescent converters to transform radiation of blue and ultra-violet diode crystals to white light.

Keywords: organic and inorganic fluorescent materials, white light emitting diodes (WLED), low pressure (LP)

1. INTRODUCTION

The use of phosphors in light sources began from fluorescent lamps (FL), in which UV radiation from LP mercury discharge is transformed to visible light by phosphor deposits on the walls of the discharge tube [1]. Twenty years ago, the concept of fluorescent transformation was also used to obtain white light from blue crystals (BC) LEDs [2]. It is clear that FL and LED lamps are closely related in terms of fluorescence.

This review is dedicated to the twenty-year long history of approaches and attempts to getting white light using energy efficient BCs and to a lesser extent, diode UV crystals (UVC) as primary radiation sources (RS).

FLs transform the consumed electric energy to UV radiation effectively, with energy efficiency greater than 64 % [3], whereas BCs are only about 50 % efficient [4]. However, transformation of LP mercury discharge UV radiation to visible radiation associated with significant energy losses. The main reason for this is Stokes shift. If exciting radiation frequency is ν_{ex}, and emitted frequency is ν_{em}, then the difference $h(\nu_{ex} - \nu_{em})$ expresses thermal losses, because $\nu_{em} < \nu_{ex}$. In FLs, frequencies of exciting mercury lines 254 and 185 nm differ from frequencies of visible radiation by several orders of magnitude, whereas, when fluorescent transforming of BC blue radiation, frequencies of exciting and emitted radiation belong to the visible spectrum and on average differ by less than 1.5 times. In the case of co-operative fluorescence (a type of anti-Stokes fluorescence), Stokes losses are absent, and energy efficiency of a near infrared laser as a primary RS is 50 %.

An optical device placed in the way of the luminous flux from the primary source of electromagnetic radiation, absorbing this radiation and emitting light by means of fluorescence, is a fluorescent converter (FC). There are two types of fluorescent crystals: fluorescent screens (FS) and fluorescent filters (FF), which differ in that FSs do not pass, and FFs pass primary RS radiation. The operational efficiency of an FC as an energy converter is determined by phosphor quantum efficiency (PQE). For FCs as optical devices, methods of exciting radiation input into the FC and of emitted radiation output are very important, but beyond the scope of this review. Methods of FC location relative to the localized primary RS, which is BC, can be divided

into two types: the FC is next to the BC (version 1), or the FC is located at a distance away from the BC (version 2).

The first attempts to create white LEDs with FCs were announced in 1997. P. Shlotter, R. Schmidt and J. Schneider introduced fluorescent green, yellow and red perylene dyes or yellow inorganic phosphorus $Y_3Al_5O_{12}$: Ce^{3+} into epoxy resin (ER) adjoining the BC [5]. As a result, white LEDs based on BCs were developed in both cases. A similar result was obtained by A. Heeger and co-workers also in 1997. They used FCs (FFs) based on a fluorescence polymer, which emitted yellow fluorescence when exciting BCs in the FC distant location version [6]. Finally, a book [7] was issued, which described the creation of BCs and subsequent work carried out by S. Nakamura et al. for many years with Nichia, including the development of white LEDs. These developments used $(Y_{l-a}Gd_a)_3(Al_{1-b}Ga_b)_5O_{12}$: *Ce* composition phosphorus as the phosphor (a, b = 0–0.5), and demonstrated how white light of different chromaticity could be obtained by changing the composition of the phosphorus. The colour coordinates (CC) of the white light obtained in [5] are unknown; and for the specimens developed in [6], they were (x and y) 0.34 and 0.29, 0.41 and 0.32, and 0.55 and 0.38 respectively. S. Nakamura's White LEDs had CCs of 0.29 and 0.30 and a luminous efficacy (LE) of 5 lm/W [7]. All of the publications mentioned above correctly predicted that LEDs with FCs would find application as light sources for illumination.

After 1997 the number of studies which sought to obtain white light suitable for illumination based on BCs, UFCs and FCs increased drastically.

2. INORGANIC PHOSPHORUS

One successful discovery by the authors of [5, 7, 8] was the use of yttrium – aluminium garnet alloyed with cerium (*YAG: Ce*), which became a basic FC material for BCs. It absorbs in the blue spectrum interval and it is steady when exposed to increased temperatures and irradiance [2, 9]. In the best specimens, it had a fluorescence quantum efficiency of more than 0.81 [10], and in commercial specimens (in 2006) the quantum efficiency was between 0.61 to 0.70 [11]. A review of literature concerning phosphorus application up to 2014 can be found in [12].

The subject of many studies is the technology of growing the phosphorus crystal and of its variants within white LEDs (casting material, powder or nanocomposite) [13–20].

In many studies, compositions were tested wherein yttrium and cerium ions were replaced with ions of other rare-earth elements [21–26]. From the *YAG: Ce* phosphorus group, especially interesting is the *Gd-YAG: Ce* composition, in which yttrium ions were partly replaced with gadolinium ions [27–29]. *Gd-YAG: Ce* has an increased thermal stability. Whilst the luminance of *YAG: Ce* fluorescence decreases by more than 60 % with increasing temperature from 25 to 205 °C, then luminance of *Gd-YAG: Ce* decreases by only 20 %. *Gd-YAG: Ce* provides a high luminous efficacy: 144 lm/W with a correlated colour temperature T_{cc} = 5639 K and 127 lm/W with T_{cc} = 4490 K.

Thermo stable phosphorus K_2TiF_6: Mn^{4+} without rare-earth elements was obtained with a red glow and quantum efficiency of 0.98 when exciting by blue radiation [30]. FC specimens based on yellow phosphorus *YAG: Ce* and on red phosphorus K_2TiF_6: Mn^{4+} operating with BC (455 nm) allowed obtaining light with a T_{cc} of 2700–2800 *K*, R_a of 83–85 and LE of 99–124 lm/W.

With a view to simplify the production technology of lighting devices based on blue crystals, the idea placing the FC at a distance is popular. Study [18] presents an FC production method with a glass ceramic disc filled with fluorescent ultra disperse powder *YAG: Ce*.

The practical outcomes of the result of all this research into FCs based on inorganic phosphorus is broad range of white LED lamps available today.

3. ORGANIC FLUORESCENT CONVERTERS

3.1. Polymeric phosphors

Materials composing a polymeric FC [6, 31] are adjoined (conjugated) polymers like polyphenylene vinylene: poly(2-methoxy-5-(2′-ethyl-hexyloxy)-1.4-phenylene vinylene) ***1***, Fig.1, poly(2- methoxy-5-(2′-ethyl- hexyloxy)-1.4-phenylene vinylene)-co-(2-butyl-5-(2′-ethyl-hexyl)-1.4-phenylene vinylene) ***2***, etc. These polymers are phosphorescent in the visible spectrum and strongly absorb light in the blue interval, where absorption factors are about 10^5 cm^{-1}. Specimens of the polymeric FCs were produced from submicronic layers of conjugated polymers, which were protect-

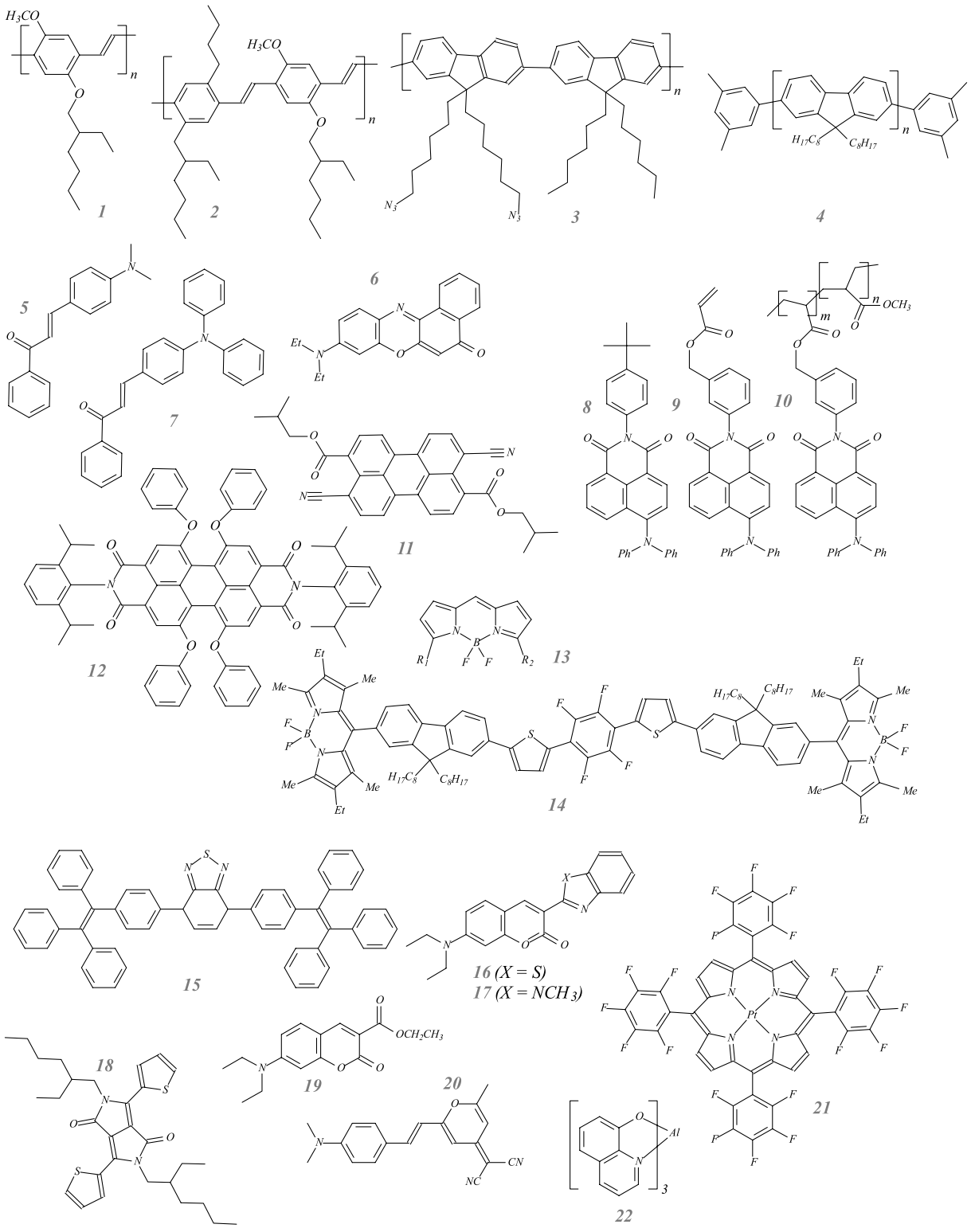

Fig. 1. Structural formulas of some phosphors

ed from the external environment by layers of glass or by encapsulation. Polymeric FCs were located at a distance from the primary RS from the outset. The best specimens had fluorescence quantum efficiency at a level of 0.6 [31, 32]. Using polymeric FCs with different BCs allowed obtaining light with colour coordinates very close to those typical for white colour. During tests lasting for more than 4000 hours, specimens of polymeric FCs showed stability no less than BCs.

Articles [33–35] are dedicated to the application of polyfluorene in white light sources. The stu-

dies [33, 34] report obtaining polyfluorene ***3*** with a molecular weight of about 20000 (n ≈ 40) and with azyid lateral groups. These groups provide interlinking of the polymeric chains. The polymer had fluorescence of white colour with a quantum efficiency of 0.86 at UV excitation. FSs of this polymer in operation had CC = 0.2554 and 0.2426, T_{cc} = 32400 K and *Ra* = 91. In [35], the FC material was a polymeric composite material: polyfluorene ***4*** with blue fluorescence and quantum points (QP) based on *CdSe/ZnS* with blue and yellow fluorescence were added to a polymethyl methacrylate (PMMA) matrix. This material was deposited directly on an UFC in specific proportions. FC specimens based on polyfluorene ***4*** and QPs with yellow fluorescence, emitted white light with T_{cc} from 3000 to 9000 *K* and with *Ra* = 85–90. In spite of the fact that quantum efficiency of polyfluorene ***4*** and of the obtained QP fluorescence was high (0.9 and 0.52 respectively), qthe uantum efficiency of the FC itself was 0.17, and LE was of about 13 lm/W. No results on FC stability were reported by [33–35].

3.2. Molecular phosphors

Several studies over the last 20 years explored the possibility of using molecular phosphors in FCs. FCs based on 4-dimethylaminochalcone ***5*** and on Nile red (7-diethylamino-3.4- benzophenoxazone ***6***) in an ER matrix were tested [36]; 4-dimethylaminochalcone ***7*** in polyethyleneglycol (PEG-6000)[37]; 4-N, N-diphenyl-9-(4-tret-butylphenyl)-1.8-naphthalimide ***8*** and fluorescein (as uranine) in ER and PMMA were tested [38]. FCs based on a copolymer of naphthalimide derivative **9** with PMMA ***10*** of the distant location type had stable characteristics within 12 days of uninterrupted operation [39]. However, the quantum efficiency of fluorescence compounds ***9*** and ***10*** were significantly lower than that of ***8*** with PMMA (0.65; 0.36 and 0.96 respectively). Studies [40–43] were dedicated to the application of yellow fluorescent *Lumogen F 083 11* dye and of red *Lumogen F 305 12* in FCs. Bor-difluoro derivative of dipyrrolmethane ***13***, namely compound ***14,*** was used in FCs for BCs in [44]. Aggregation phosphor 4.7-bis [4-(1,2,2- triphenylvinyl) phenyl] benzo-2,1,3- thiadiazole ***15*** was used in study [45].

Coumarine – 6 ***16***, coumarine 30 ***17*** and N-alkylated dipyrrolopyrrol with thiophen substitutes ***18***, as well as 7 (diethylamine) – coumarine-3-carbonic acid ***19*** and 4-(dicyanomethylene)- 2-methyl-6-(4-dimethylaminosteral) – 4H-pyran 20 were the basis of the FC in [46–48].

A noticeable degradation of phosphors in the listed publications was observed during operation from 10 min [45] up to 10 days [48].

3.3. Coordination compounds

Metal-porphyrines and coordinated compounds of metals are prospective materials for optoelectronics. In [49], their possible application in FCs is illustrated by using platinum (II) *meso*-tetrakis (pentafluorophenyl) porphyrine ***21*** and tris (8–8-oxyquinolinate) of aluminium (III) ***22***. The fluorescence of compound ***21*** in a PC matrix did not change at 120 °C during 1000 hours of operation. The intensity of fluorescence ***21*** decreased by one third after UV radiation with irradiance of 10 W/m^2 during 100 hours. UFC radiation with a certain concentration of compounds ***21*** and ***22*** transforms into white with a CC of 0.32 and 0.31, FI = 90.6, T_{cc} = 6800 K at LE =10 lm/W.

3.4. Quantum points

The first explorations of the possibility of using quantum points (QP) in FCs for LEDs are presented in [50, 51]. In [50], white light was obtained by a combination of blue fluorescence of an organic polymer with green and red fluorescence of *CdSe* of two types (3 and 7 nm in size), and in [51] *CdSe* QPs of a single type (1.5 nm in size) was used. The QPs synthesised in [51] had a wide radiation spectrum, an expressed edge in the long-wave absorption band and a relatively large Stokes shift. The authors reported on 10 days of operation stability but the material fluorescence quantum efficiency was very low, about 0.02. In nanotechnology literature properties of QP fluorescence describe quite opposite parameters: a narrowness of the radiation spectrum, a wide absorption band and a high quantum efficiency. The “narrow-band” QPs are soon likely to become the fluorescent material replacing technologies based on organic LEDs in colour displays (*Colour IQ* system by *QD Vision*). Meanwhile “broadband” QPs have not successfully increased their quantum efficiency to an acceptable level.

An example of successful use of the “narrow-band” QPs with yellow fluorescence in FCs for BCs can be found in study [52]. It was achieved using colloidal QPs with a core of *Cu-In-S* material of

Cu/In = ¼ relation, with two *ZnS* protective shells. The QP core size is 2.72 nm. These QPs had a high fluorescence quantum efficiency of 0.92–0.97. An SiO_x polymer was used to protect the QP layer from O_2 penetration and from its destuctive effect.

3.5. Frame metal-organic compounds

Frame metal-organic compounds (FMC) are a new class of crystal materials, which consist of transitional metal and of polydentate organic ligand cations.

A specific feature of the frame metal-organics crystal structures is the presence of microscopic pores or channels, where guest molecules can be located.

FMCs are attractive as fluorescent materials as they present the possibility of using inorganic and organic elements in fluorescence centres or to add phosphor molecules to the FMC structure as guests placing them into the pores. Results of studies using FMC in FCs are given in articles [53–56]. For the present, the highest quantum efficiency level of white FMC fluorescence was 0.2, reported in [56]. White fluorescence at UV excitation has become a result of the addition of matrix blue fluorescence with yellow fluorescence of an iridic system: $Ir(ppy)_2(bpy)]^+$ (*ppy* – 2-phenylpyridine, *bpy* – 2,2′- bipyridine) placed in the host pores. The quantum efficiency of the material fluorescence decreased by 10 % at 150 °C, which is an indicative of its high thermal stability. The photostability of the material was not reported.

4. CONCLUSION

1. At present, inorganic phosphorous is the only fluorescent material class successfully applied in FCs for BCs and UFCs in the field of illumination.

2. Among organic phosphors, polymeric phosphors are most likely to be used in future FCs.

3. An insurmountable obstacle for the use of molecular organic phosphor in FCs is the photo degradation of phosphors at comparatively high levels of absorbed energy, which increases with the influence of oxygen. Nevertheless, these materials are of interest for lighting engineering, for example, in decorative and design use and in outdoor light advertising.

4. The photochemical stability of new fluorescent materials, to which coordination compounds QP and FMC belong, is insufficiently understood and their prospects for application in FCs are not clear.

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