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AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO,
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KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD,
ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI,
NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU,
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(54) Title: LUMINESCENT MATERIAL AND LIGHT EMITTING DEVICE COMPRISING SUCH LUMINESCENT MATERIAL

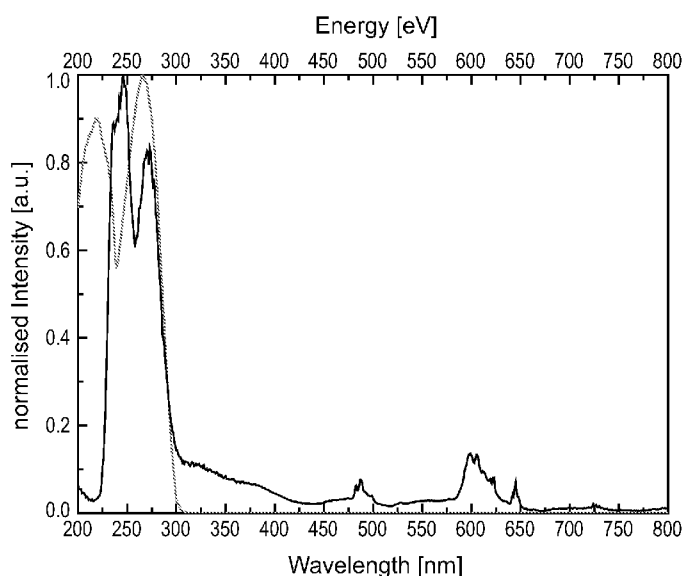


FIG. 3

(57) Abstract: The invention provides a luminescent material comprising a component selected from the group comprising $(Y_{1-x}Lu_x)_9LiSi_6O_{26}:Ln$ or/and $AE_5(PO_4)_3F:Ln,A$, wherein Ln is a trivalent rare earth metal, AE is a divalent alkaline earth metal, and A is a monovalent alkaline metal, $x > 0.0$ and < 1.0 . The luminescent material has an emission peak in the UV-C range when being excited by light in the UV spectrum range. The invention further provides a light emitting device comprising the said luminescent material and a method of using said light emitting device for disinfection or purification of air, water or surfaces.



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LUMINESCENT MATERIAL AND LIGHT EMITTING DEVICE COMPRISING SUCH LUMINESCENT MATERIAL

5 FIELD OF THE INVENTION

The present invention relates to luminescent material, especially to the field of luminescent material for light emitting devices emitting UV radiation.

BACKGROUND OF THE INVENTION

10 UV radiation sources have found many application areas, such as spectroscopy, cosmetic skin treatment, medical skin treatment, disinfection or purification of water and air, polymer hardening, photochemistry, surface curing, and wafer processing.

Many of the above mentioned application areas require deep UV radiation, i.e. UV-C (200 – 280 nm) or even VUV radiation (100 – 200 nm), wherein fast switching cycles and invariance
15 against temperature changes are desired features.

Low-pressure Hg discharge lamps are currently widely used as UV radiation sources and they have an emission spectrum which is dominated by two lines, viz. at 185 and 254 nm. However, increasing the Hg vapor pressure may result in an almost continuous spectrum extending from the deep UV to the deep red spectral range. Moreover, the application of Hg implies a rather
20 strong dependence on temperature and sensitivity to fast switching cycles.

For more than 10 years, the application of dielectric barrier (DB) noble gas excimer discharge has been regarded as an alternative discharge concept for the development of UV emitting radiation sources. The Xe excimer discharge, e.g., emits mainly 172 nm radiation and DB driven quartz lamps comprising Xe as a filling gas show a wall plug efficiency of more than
25 30%. Quartz lamps based on a Xe excimer discharge are widely used for the cleaning of wafer surfaces due to the sufficiently high energy of the emitted 172 nm (VUV) photons to cleave any type of organic bonds. Fluorescent Xe excimer discharge lamps using one or several VUV

to UV-C down-converting phosphors are of particular interest for disinfection or purification purposes.

Presently applied UV luminescent materials for these Xe, Ne, or Xe/Ne excimer lamps still have a couple of drawbacks, for example, including:

- 5 - low conversion efficiency
- low photochemical stability
- low chemical stability
- low spectral overlap with the germicidal action curve.

10 Therefore there is a need to develop alternative luminescent materials for converting UV radiation from for instance fluorescent Xe excimer discharge lamps into radiation spectra that can more properly be used for instance in disinfection or purification areas.

SUMMARY OF THE INVENTION

15 It is an object of the present invention to provide alternative luminescent materials for light emitting devices emitting UV radiation.

It is another object of the present invention to provide a light emitting device comprising luminescent materials, which device shows intense and efficient UVC emission with a spectral power distribution that fits well to the germicidal action spectrum.

20 It is yet another object of the present invention to provide a system comprising a light emitting device, which system can use the light emitted by the light emitting device to disinfect or purify air, or water, etc.,.

25 According to an embodiment of the present invention, a luminescent material is provided that comprises a component selected from the group comprising $(Y_{1-x}Lu_x)_9LiSi_6O_{26}:Ln$ or/and $AE_5(PO_4)_3F:Ln,A$, wherein Ln is a trivalent rare earth metal, AE is a divalent alkaline earth metal, and A is a monovalent alkaline metal, $x \geq 0.0$ and ≤ 1.0 .

According to a preferred embodiment, Ln is selected from the group comprising trivalent Pr,

Nd or mixtures thereof. AE is selected from the group comprising divalent Ca, Sr, Ba or mixtures thereof. A is selected from the group comprising monovalent Li, Na, K, Rb, Cs or mixtures thereof.

5 The luminescent material has an emission peak in the UVC (i.e. 200-280nm) range when being excited by light with an excitation spectrum in the UV spectrum range, preferably in the VUV or UVC range. Light with such an excitation spectrum can be achieved using a Hg or noble gas discharge lamp, for instance, amalgam lamps with an emission peak at around 185nm, low-pressure Hg discharge lamps with an emission peak at around 254nm, medium-pressure Hg discharge lamps with an emission peak at around 265nm, and Xe, Ne, or Xe/Ne excimer lamps
10 with an emission peak at around 172nm. Alternatively, newly developed LED lamps, like (Al,Ga)N LED lamps, or other types of existing lamps, and even some new types of lamps yet to be developed, can be used as the light source to provide the excitation spectrum, as long as such lamps can emit a proper excitation spectrum needed for the luminescent material to emit the UV-C.

15 Surprisingly, it has been found that the above proposed luminescent materials show intense and efficient UV-C emission with a spectral power distribution that fits well to the germicidal action spectrum.

According to another embodiment of the present invention, a light emitting device is provided which is capable of emitting a first light in a first UV spectrum range, and comprises at least
20 one of the above proposed luminescent materials to absorb at least part of the first UV light and to emit a second light in a second UV spectrum range different from the first UV spectrum range.

It has been found that such a light emitting device has, for a wide range of applications, especially for germicide application, at least one of the following advantages:

- 25
- Improved efficacy due to the optimized emission spectrum with respect to the action curve of the application and due to less re-absorption by the luminescent materials;
 - Improved stability of the UVC output and thus improved operational lifetime of the light emitting device;
 - smaller dependence of the efficacy on temperature.

According to a preferred embodiment, the light emitting device comprises a discharge lamp provided with a discharge vessel comprising a gas filling having a discharge-maintaining composition, and at least a part of a wall of the discharge vessel is coated with the luminescent material. Alternatively, the discharge lamp comprises a Hg or noble gas discharge lamp.

5 Alternatively, the light emitting device comprises a newly developed LED lamp like a (Al, Ga)N LED lamp, or an already existing lamp type, or even a new type of lamp yet to be developed. For LED lamps, the luminescent materials can be configured as a dome to cover the LED chips, or to be coated on an optical component like a lens or bulb.

10 According to another embodiment of the present invention, a system comprising at least one of the above proposed light emitting devices is provided, the system further comprising a unit capable of making the light emitted by the light emitting device irradiate an object to be sterilized. This system can be used in germicide applications via photochemical processing with the help of the light emitted by the light emitting device, for instance in disinfection or purification of air, water or surfaces. Such a unit, for example, can be a light guiding means to
15 transport the light from the light emitting device to a surface so that the light can directly irradiate the surface to sterilize said surface. Alternatively, such a unit may comprise a suction device configured to draw certain air into the system so that the light can directly irradiate the air for purification thereof.

20 According to another embodiment of the present invention, a germicide application method is also provided, which comprises the step of making the light emitted by at least one of the above proposed light emitting devices irradiate an object to be sterilized.

Alternatively, the method can be used in disinfection or purification of air, water or surfaces. Thus, by making the light emitted by the above mentioned light emitting device irradiate air, water or a surface, the air, water or surface can be sterilized.

25 It has been found that the proposed system and method have a good germicidal effect due to the UV-C emission having a spectral power distribution that fits well to the germicidal action spectrum.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other objects and features of the present invention will become apparent from the following detailed description of the various aspects of embodiments with reference to the accompanying drawings.

- 5 Fig. 1 shows an XRD pattern of a first exemplary luminescent material according to the present invention (Example I: $Ca_5(PO_4)_3F:Pr^{3+}(1\%)Na^+(1\%)$);
- 10 Fig. 2 shows the excitation spectrum (left spectrum), emission spectrum (right spectrum) and reflection spectrum (upper right spectrum) of the first luminescent material according to the present invention (Example I);
- Fig. 3 shows a comparison between the emission spectrum of the first luminescent material (Example I) and the desired spectrum of the germicidal action;
- 15 Fig. 4 shows an XRD pattern of a second exemplary luminescent material according to the present invention (Example II: $Sr_5(PO_4)_3F:Pr^{3+}(1\%)Na^+(1\%)$);
- Fig. 5 shows the excitation spectrum (left spectrum), the emission spectrum (right spectrum) and the reflection spectrum (upper right spectrum) of the second luminescent material according to the present invention (Example II);
- 20 Fig. 6 shows a comparison between the emission spectrum of the second luminescent material (Example II) and the desired spectrum of the germicidal action curve;
- 25 Fig. 7 shows an XRD pattern of a third exemplary luminescent material according to the present invention (Example III: $Y_9LiSi_6O_{26}:Pr^{3+}(1\%)$);
- Fig. 8 shows the excitation spectrum (left spectrum), the emission spectrum

(right spectrum) and the reflection spectrum (upper right spectrum) of the third luminescent material according to the present invention (Example III);

Fig. 9 shows an XRD pattern of a fourth exemplary luminescent material according to the present invention (Example IV:

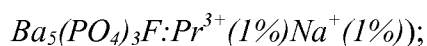


Fig. 10 shows the excitation spectrum (left spectrum), the emission spectrum (right spectrum) and the reflection spectrum (upper right spectrum) of the fourth luminescent material according to the present invention (Example IV).

DETAILED DESCRIPTION OF EMBODIMENTS

The detailed description of the embodiments given below will mainly focus on examples of luminescent materials. As for the light emitting device, the system and the method proposed in the present invention, the prior part has given a useful description and reference can be made to the existing relevant papers or products.

Example I:

Example I refers to $\text{Ca}_5(\text{PO}_4)_3\text{F}:\text{Pr}^{3+}(1\%)\text{Na}^+(1\%)$, which can be made in the following way:

The starting materials 1.009 g CaCO_3 , 4.0004 g $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, 0.32 g nanoscale CaF_2 , and 0.076 g PrF_3 und 0.016 g NaF have been milled for 0.5 hours. The blend has been subsequently annealed at around 1100 °C under Nitrogen for 1 hour. Finally, the material is milled and sieved through a 36 µm sieve.

Fig. 1 shows an XRD pattern of the material of Example I. Fig. 2 shows the excitation spectrum (left spectrum), the emission spectrum (right spectrum) and the reflection spectrum (upper right spectrum) of the material of Example I. Fig. 3 shows a comparison between the emission spectrum (the curve with relatively narrow extension along the wavelength in the drawing, as well as in other drawings of the same type referred to below) of the material of

Example I and the desired spectrum of the germicidal action. The emission maximum of $\text{Ca}_5(\text{PO}_4)_3\text{F}:\text{Pr},\text{Na}$ is at around 245 nm, which shows a good overlap with the germicidal action curve. It can clearly be seen that this material is an excellent material for use in discharge lamps for UV-C radiation.

5

Example II

Example II refers to $\text{Sr}_5(\text{PO}_4)_3\text{F}:\text{Pr}^{3+}(1\%)\text{Na}^+(1\%)$, which can be made in the following way:

The starting materials 5.036 g SrCO_3 , 2.675 g $(\text{NH}_4)_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$, 0.487 g nanoscale SrF_2 , and 0.076 g PrF_3 und 0.016 g NaF have been milled for 0.5 hours. The blend has been subsequently
10 annealed at around 1100 °C under Nitrogen for 1 hour. Finally, the material is milled and sieved through a 36 µm sieve.

The emission maximum of $\text{Sr}_5(\text{PO}_4)_3\text{F}:\text{Pr},\text{Na}$ is at about 240 nm, which also shows a good overlap with the germicidal action curve. It can clearly be seen from Figs. 4-6 that this material is an excellent material for use in discharge lamps for UV-C radiation.

15

Example III:

Example III refers to $\text{Y}_9\text{LiSi}_6\text{O}_{26}:\text{Pr}^{3+}(1\%)$, which can be made in the following way:

The starting materials 4.000 g Y_2O_3 , 0,147 g Li_2CO_3 , 1,433 g nanoscale SiO_2 , and 0.061 g Pr_6O_{11} are suspended in ethanol and the material is ground until the solvent has completely
20 evaporated. Afterwards, the dried material is fired at 1000 °C under CO for 6 hours and subsequently ground and fired at 1100 °C under CO for 6 hours. Finally, the material is milled and sieved through a 36 µm sieve. It can clearly be seen from Figs. 7-8 that this material is an excellent material for use in discharge lamps for UV-C radiation.

25

Example IV:

Example IV refers to $\text{Ba}_5(\text{PO}_4)_3\text{F}:\text{Pr}^{3+}(1\%)\text{Na}^+(1\%)$, which can be made in the following way:

The starting materials 5.036 g BaCO_3 , 2.675 g $(\text{NH}_4)_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$, 0.487 g nanoscale BaF_2 , and 0.076 g PrF_3 und 0.016 g NaF have been milled for 0.5 hours. The blend has been subsequently annealed at 1100 °C under Nitrogen for 1 hour. Finally, the material is milled and sieved through a 36 μm sieve. It can clearly be seen from Figs. 9-10 that this material is an excellent material for use in discharge lamps for UV-C radiation.

The embodiments described above are merely preferred embodiments of the present invention. Other variations of the disclosed embodiments can be understood and effected by those skilled in the art in practicing the claimed invention, from a study of the drawings, the disclosure, and the appended claims. These variations shall also be considered to be within the scope of the present invention. In the claims and description, use of the verb "comprise" and its conjugations does not exclude other elements or steps, and the indefinite article "a" or "an" does not exclude a plurality.

1. A luminescent material comprising a component selected from the group comprising $(Y_{1-x}Lu_x)_9LiSi_6O_{26}:Ln$ or/and $AE_5(PO_4)_3F:Ln,A$, wherein Ln is a trivalent rare earth metal, AE is a divalent alkaline earth metal, and A is a monovalent alkaline metal, $x \geq 0.0$ and ≤ 1.0 .

2. The luminescent material according to claim 1, wherein Ln is selected from the group comprising trivalent Pr, Nd or mixtures thereof.

3. The luminescent material according to claim 1, wherein AE is selected from the group comprising divalent Ca, Sr, Ba or mixtures thereof.

4. The luminescent material according to claim 1, wherein A is selected from the group comprising monovalent Li, Na, K, Rb, Cs or mixtures thereof.

5. The luminescent material according to claim 1, wherein the luminescent material has an emission peak in the UVC range when being excited by light with an excitation spectrum in the UV spectrum range.

6. A light emitting device, which is capable of emitting a first light in a first UV spectrum range, comprising a luminescent material according to any one of claims 1 to 5 to absorb at least part of the first UV light and to emit a second light in a second UV spectrum range different from the first UV spectrum range.

7. The light emitting device according to claim 6, wherein the light emitting device comprises a discharge lamp, provided with a discharge vessel comprising a gas filling with a

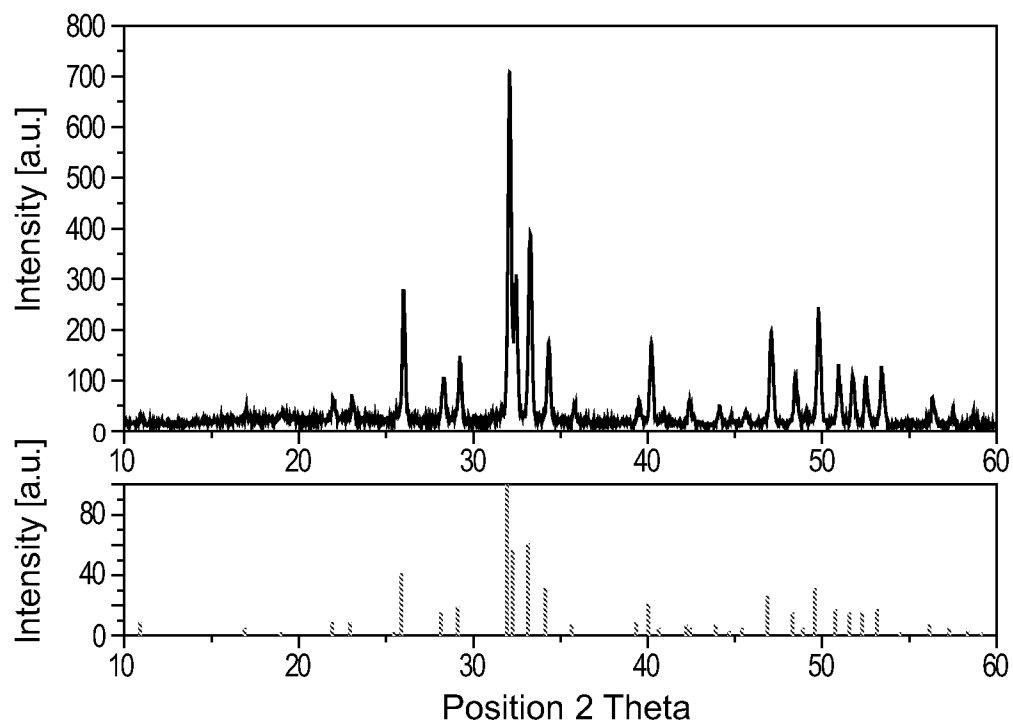
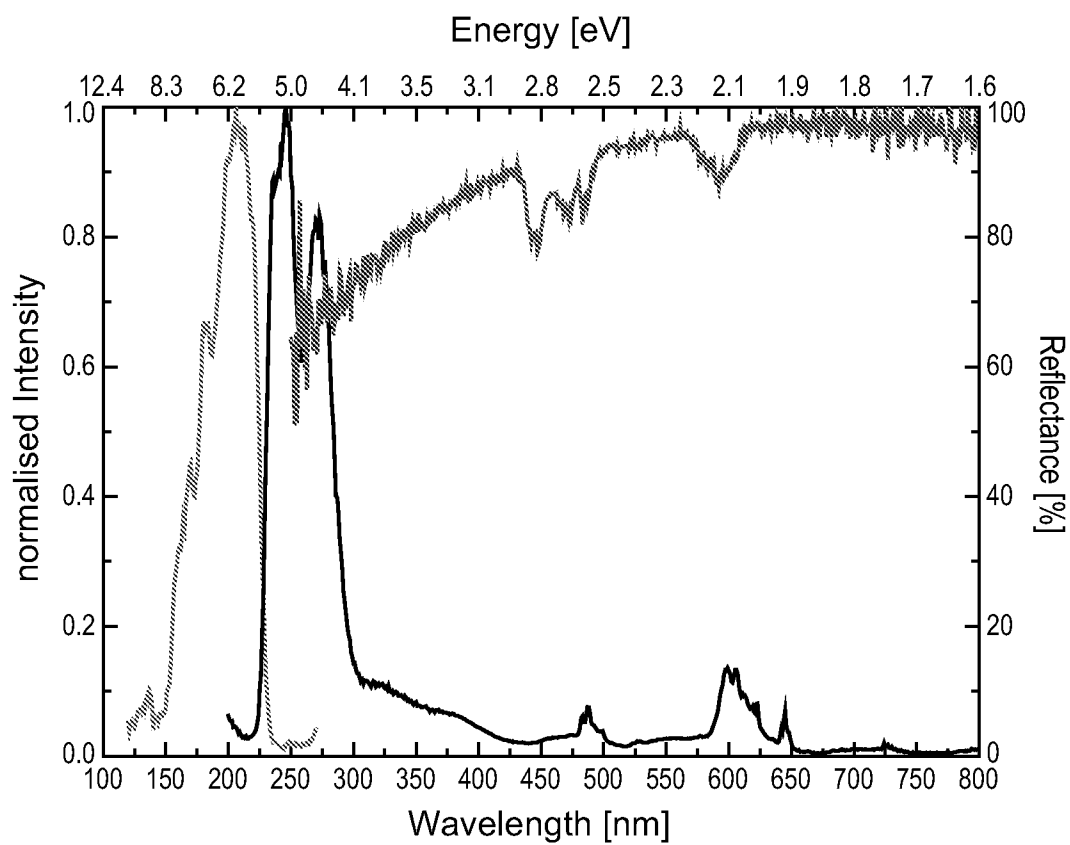
discharge-maintaining composition, and at least a part of a wall of the discharge vessel is coated with the luminescent material.

8. The light emitting device according to claim 6, wherein the discharge lamp comprises a Hg or noble gas discharge lamp, or/and a LED lamp.

9. A system comprising a light emitting device according to any one of claims 6 to 8, the system further comprising a unit capable of making the light emitted by the light emitting device irradiate an object to be sterilized.

10. A method of applying a germicidal agent, comprising the step of making the light emitted by a light emitting device according to any one of claims 6 to 8 irradiate an object to be sterilized.

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**FIG. 1****FIG. 2**

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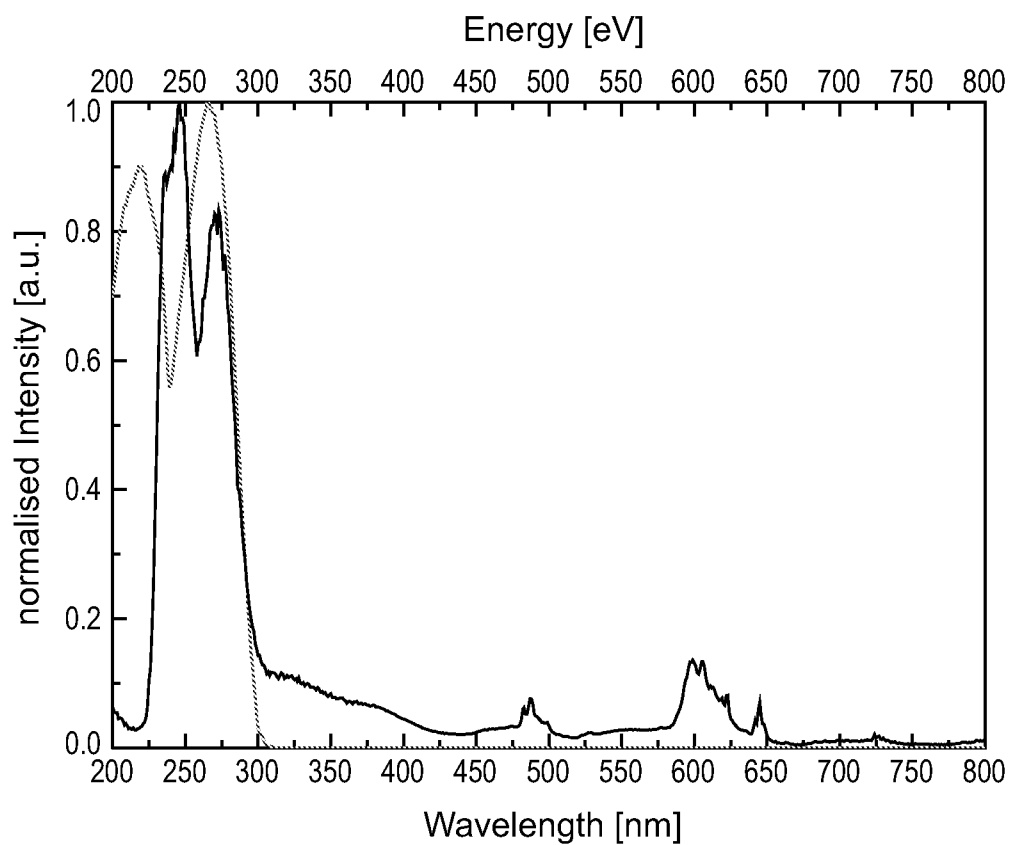


FIG. 3

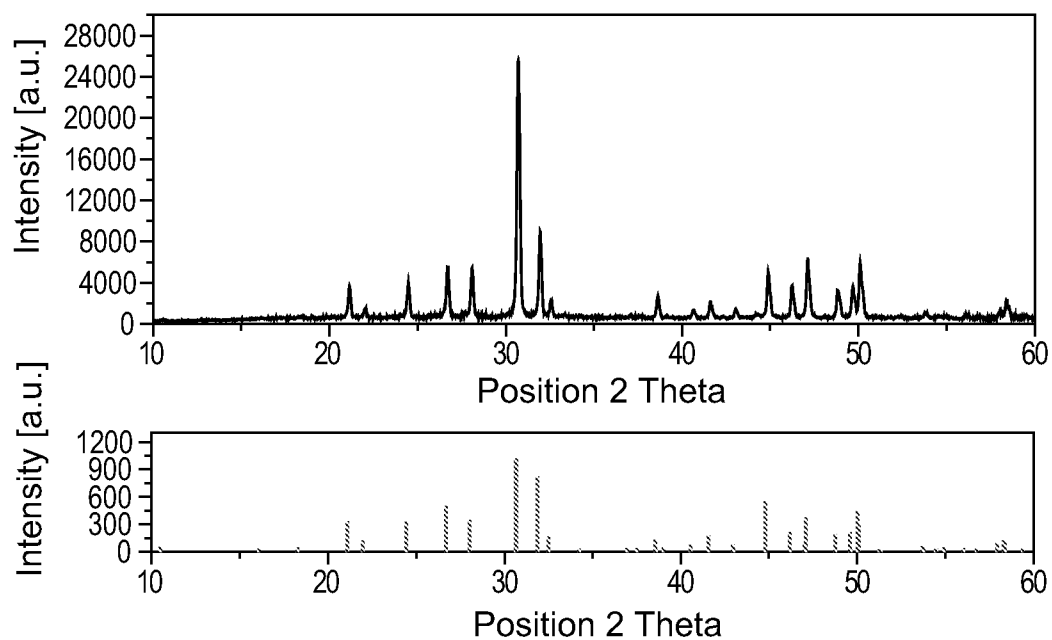
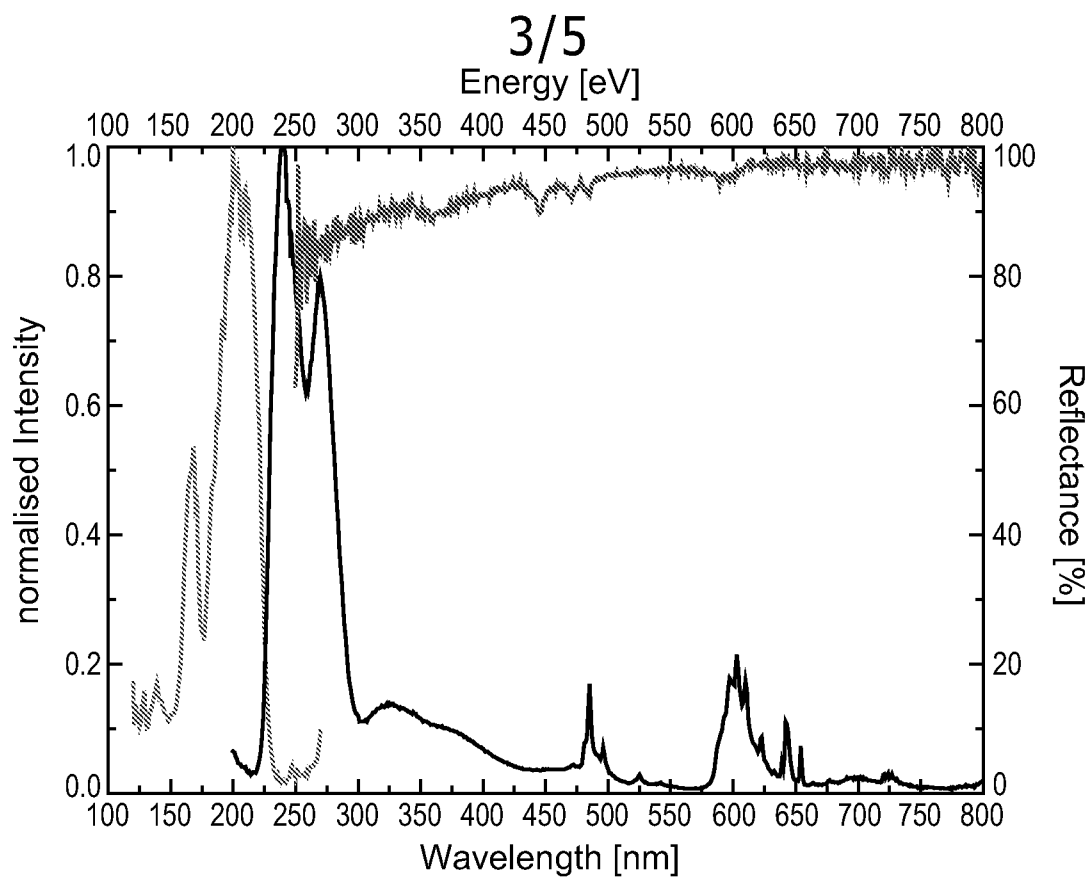
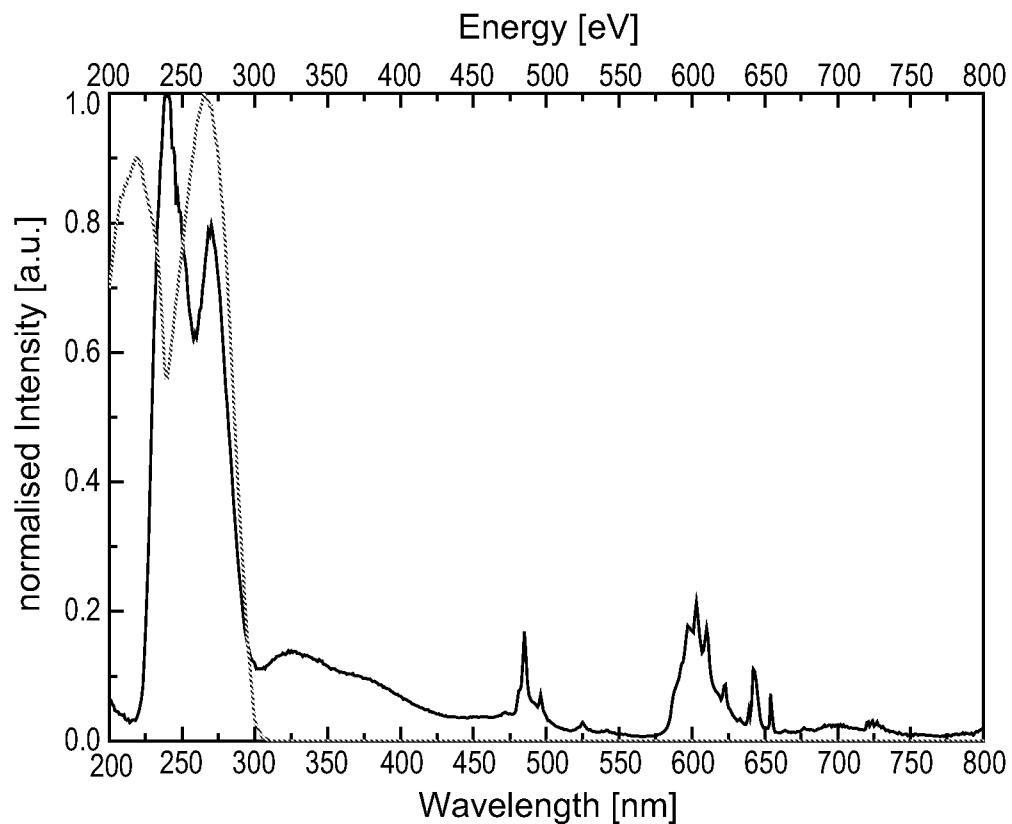


FIG. 4

**FIG. 5****FIG. 6**

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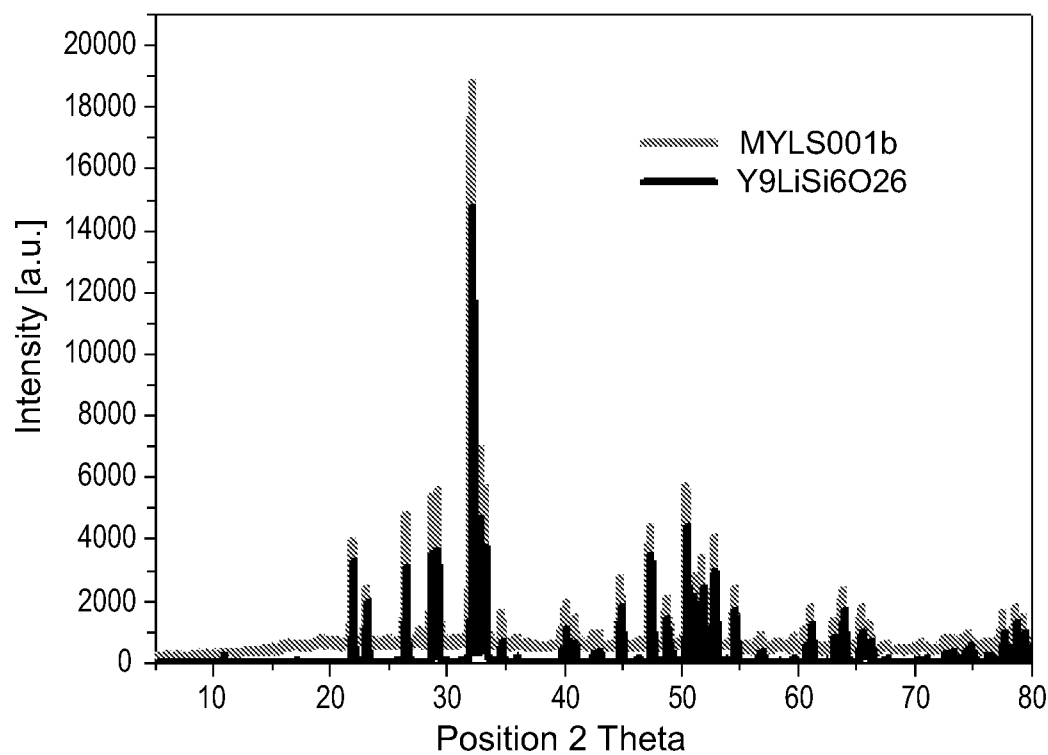


FIG. 7

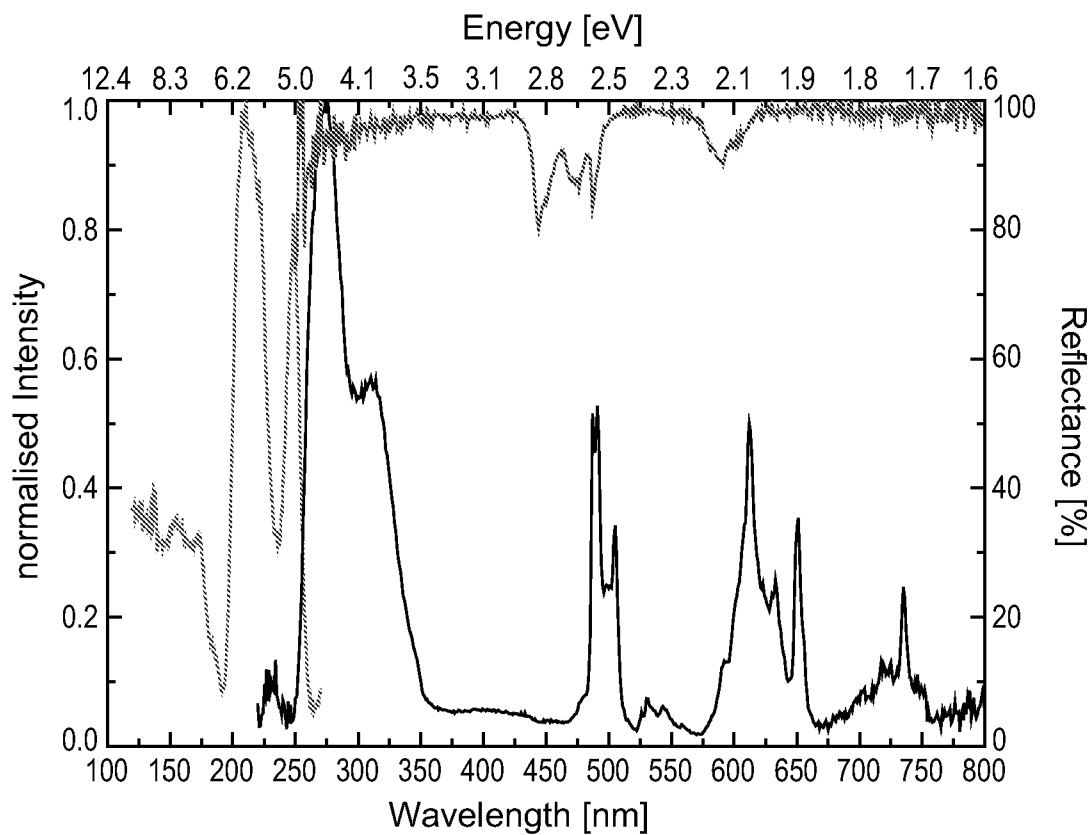


FIG. 8

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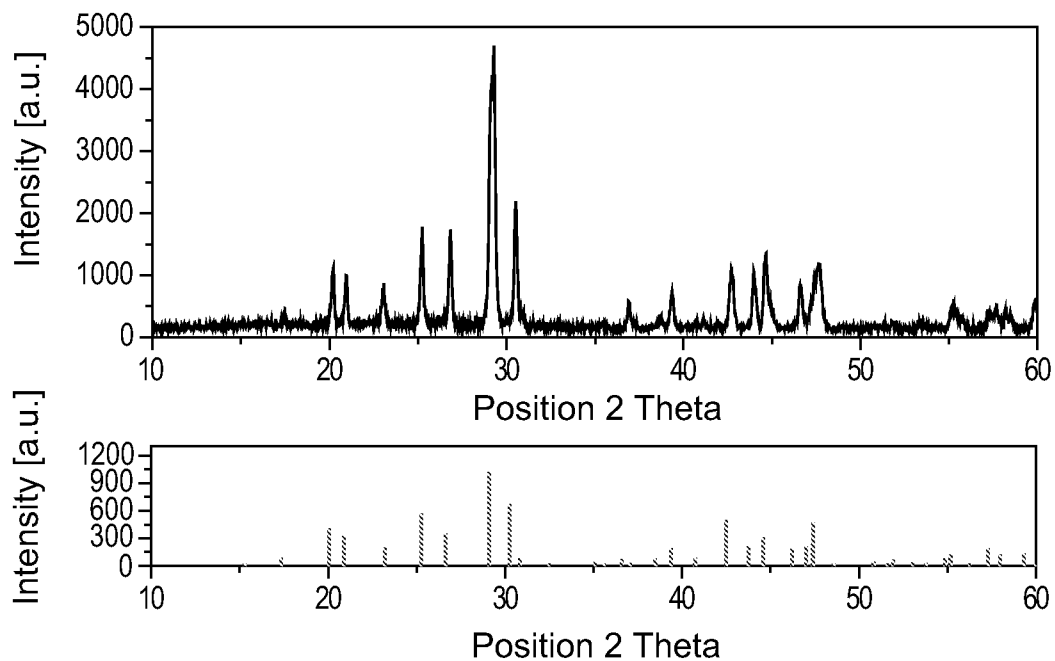


FIG. 9

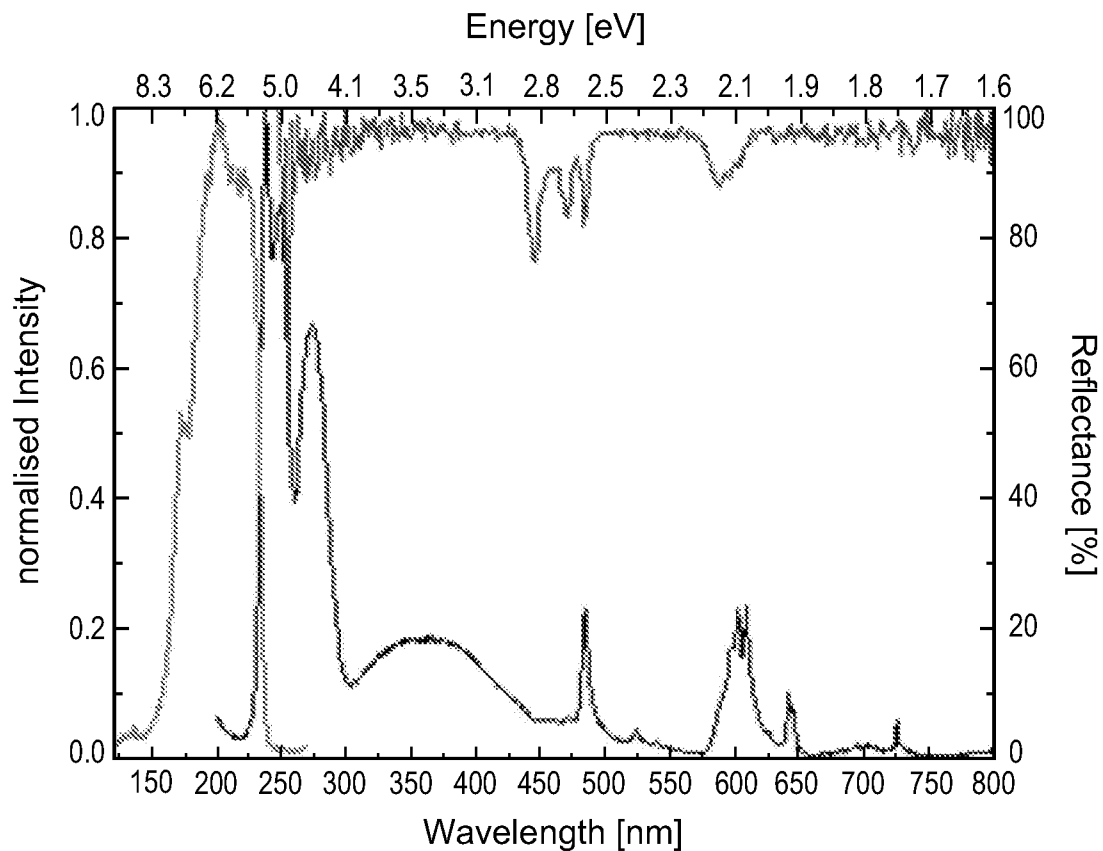


FIG. 10

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2011/054589

A. CLASSIFICATION OF SUBJECT MATTER
INV. C09K11/73 C09K11/79 H01J61/44
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2009/140630 A1 (KIJIMA NAOTO [JP] ET AL) 4 June 2009 (2009-06-04)	1,5
A	paragraphs [0001], [0665] -----	2
X	WO 2005/042812 A1 (ZAGUMENNYI ALEXANDER IOSIFOVIC [RU]; ZAVARTSEV YURI DMITRIEVICH [RU];) 12 May 2005 (2005-05-12)	1,5
A	page 12, lines 10-14 -----	2
X	EP 1 842 892 A1 (OSRAM SYLVANIA INC [US]) 10 October 2007 (2007-10-10)	6-10
A	paragraphs [0004], [0005], [0052], [0053] -----	1-5



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

5 December 2011

Date of mailing of the international search report

21/03/2012

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
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Authorized officer

Delaporte, P

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2011/054589

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1, 2, 5-10(all partially)

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1, 2, 5-10(all partially)

A luminescent material comprising a component $(Y_{1-x}Lu_x)_9LiSi_6O_{26}:Ln$, wherein Ln is a trivalent rare earth metal, $x \geq 0.0$ and ≤ 1.0 (independent claim 1 in part and claims 2 and 5 in part depending thereon), a light emitting device which is capable of emitting a first light in a first UV spectrum range, comprising a luminescent material according to any one of claim 1 in part, claim 2 in part or claim 5 in part to absorb at least part of the first UV light and to emit a second light in a second UV spectrum range different from the first UV spectrum range (independent claim 6 in part and claims 7 and 8 in part depending thereon), a system comprising a light emitting device according to any one of claims 6 to 8 in part, the system further comprising a unit capable of making the light emitted by the light emitting device irradiate an object to be sterilized (independent claim 9 in part) and a method of applying a germicidal agent, comprising the step of making the light emitted by a light emitting device according to any one of claims 6 to 8 in part irradiate an object to be sterilized (independent claim 10 in part).

2. claims: 1-10(partially)

A luminescent material comprising a component $AE_5(P_4O_4)_3F:Ln,A$, wherein Ln is a trivalent rare earth metal, AE is a divalent alkaline earth metal, and A is a monovalent alkaline metal (independent claim 1 in part and claims 2-5 in part depending thereon), a light emitting device which is capable of emitting a first light in a first UV spectrum range, comprising a luminescent material according to any one of claims 1-5 in part to absorb at least part of the first UV light and to emit a second light in a second UV spectrum range different from the first UV spectrum range (independent claim 6 in part and claims 7-8 in part depending thereon), a system comprising a light emitting device according to any one of claims 6 to 8 in part, the system further comprising a unit capable of making the light emitted by the light emitting device irradiate an object to be sterilized (independent claim 9 in part) and a method of applying a germicidal agent, comprising the step of making the light emitted by a light emitting device according to any one of claims 6 to 8 in part irradiate an object to be sterilized (independent claim 10 in part).

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2011/054589

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