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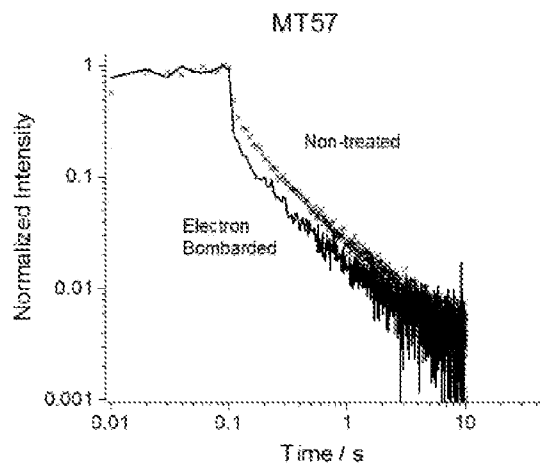
(54) Keksinnön nimitys - Uppfinningens benämning - Title of the invention
MENETelmä LUMINESOIVAN MATERIAALIN ELEKTRONIPOMMITAMISEKSI
Förfarande för elektronbombardering av luminescerande material
A METHOD FOR ELECTRON BOMBARDMENT OF LUMINESCENT MATERIAL

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Byron, H. et al. Journal of Alloys and Compounds, March 26, 2021, vol 872, 159671., EP 3455167 B1, GB 1187982 A, Shidlovsky, I. et al. Solid State Communications, June 4, 1975, vol 18, pp. 155-158., US 3799881 A

(57) Tiivistelmä - Sammandrag - Abstract

Esillä oleva keksintö koskee menetelmää materiaalin, jota edustaa $(M')_8(M''M''')_6O_{24}(X,X')_2M''''$, käsittelemiseksi elektronipommituksella tyhjiökammiossa, jolla on paine, joka mahdollistaa elektronisuihkun muodostuksen. Lisäksi keksintö koskee menetelmää tällaisen materiaalin optisesti simuloitun luminesenssin intensiteetin lisäämiseksi sekä menetelmää tällaisen materiaalin jälkiloisteen vähentämiseksi. Edelleen esillä oleva keksintö koskee menetelmää tietokoneradiografiasta, tietokonetomografiasta ja digitaalisesta radiografiasta valitun kuvan kontrastin lisäämiseksi.

The present invention concerns a method for treating a material represented by $(M')_8(M''M''')_6O_{24}(X,X')_2M''''$, by electron bombardment in a vacuum chamber having a pressure enabling formation of an electron beam. In addition, the invention concerns a method of increasing intensity of optically simulated luminescence of such material as well as a method of decreasing afterglow of such material. Further, the present invention concerns a method for increasing contrast of an image selected from computed radiography, computed tomography and digital radiography.



A METHOD FOR ELECTRON BOMBARDMENT OF LUMINESCENT MATERIAL

FIELD

The present invention relates to luminescent materials and their treatment by electron bombardment. The present invention also relates to a method for increasing intensity of optically stimulated luminescence of such materials, and to a method for decreasing afterglow of such materials. Further, the invention relates to a method for increasing contrast of an image selected from computed radiography, computed tomography and digital radiography, the material used for imaging comprising the present material.

BACKGROUND AND OBJECTS

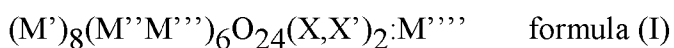
Hackmanite, which is a variety of sodalite material, is a natural mineral having the chemical formula of $\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}(\text{Cl},\text{S})_2$. A synthetic hackmanite-based material can be prepared and can also be called hackmanite. These synthetic materials are described for example in WO 2017/194825 and WO 2017/194834, and can be used for various devices, such as for detecting and indicating the intensity of a radiation (as described in WO 2019/092309) or for determining the amount of radiation (as described in WO 2019/092308).

These materials are used also for example in computed radiography (CR). However, a problem with these types of materials is that their afterglow after optically stimulated luminescence (OSL) decreases the sharpness of the radiography images. It is thus an aim of the present disclosure to provide a method for decreasing this afterglow to increase sharpness of the images. A further aim is to increase the OSL intensity when the material is excited with laser, again to increase the readability of the computed radiography images. In general, it is an aim of the present disclosure to improve readability (i.e. sharpness and intensity of signal) of computed radiography images and similar.

SUMMARY OF THE INVENTION

The invention is defined by the features of the independent claims. Some specific embodiments are defined in the dependent claims.

According to a first aspect, there is provided a method for treating a luminescent material by electron bombardment. In particular, a material represented by formula (I) is treated by electron bombardment



5 wherein

- M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations;
 - M'' represents a trivalent monoatomic cation of an element selected from Group 13 of the IUPAC periodic table of the elements, or of a transition element selected from any of
10 Groups 3 – 12 of the IUPAC periodic table of the elements, or any combination of such cations;
 - M''' represents a monoatomic cation of an element selected from Group 14 of the IUPAC periodic table of the elements, or of an element selected from any of Groups 13 and 15 of the IUPAC periodic table of the elements, or of Zn, or any combination of such
15 cations;
 - X represents an anion of an element selected from Group 17 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X is absent;
 - X' represents an anion of one or more elements selected from Group 16 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X' is absent;
20 and
 - M'''' represents a dopant cation of an element selected from rare earth metals of the IUPAC periodic table of the elements, or from transition metals of the IUPAC periodic table of the elements, or of Ba, Sr, Tl, Pb, or Bi, or any combination of such cations, or wherein M'''' is absent;
- 25 with the proviso that at least one of X and X' is present,
the electron bombardment being carried out in a vacuum chamber having a pressure enabling formation of an electron beam.

According to a second aspect, there is provided a method for increasing intensity of optically simulated luminescence of a material represented by formula (I), by carrying out
30 the electron bombardment treatment described above.

According to a third aspect, there is provided a method for decreasing afterglow of a material represented by formula (I), by carrying out the electron bombardment described above.

According to a fourth aspect, there is provided a method for increasing contrast of an image selected from computed radiography, computed tomography and digital radiography, by using the electron bombarded material of formula (I) as defined above, treated with the method as described above.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 illustrates the decrease of optically stimulated luminescence afterglow in electron bombarded material of formula (I) according to one embodiment compared to non-treated material.

Figure 2 illustrates the decrease of optically stimulated luminescence afterglow in electron bombarded material of formula (I) according to another embodiment compared to non-treated material.

Figure 3 illustrate the decrease of optically stimulated luminescence afterglow in electron bombarded material of formula (I) according to a further embodiment compared to non-treated material.

Figure 4 illustrates the decrease of normal afterglow in electron bombarded material of formula (I) according to one embodiment compared to non-treated material.

Figure 5 illustrates the decrease of normal afterglow in electron bombarded material of formula (I) according to another embodiment compared to non-treated material.

Figure 6 illustrates the increase of intensity of optically stimulated luminescence in electron bombarded material according to an embodiment.

Figure 7 illustrates the increase of intensity of optically stimulated luminescence in electron bombarded material according to a further embodiment.

Figure 8 illustrates the increase of intensity of optically stimulated luminescence in electron bombarded material according to another embodiment.

Figure 9 illustrates the effect of the time of electron bombardment on the intensity of optically stimulated luminescence according to an embodiment.

Figure 10 illustrates the effect of the time of electron bombardment on the intensity of optically stimulated luminescence according to the embodiment of Figure 9.

Figure 11 illustrates the effect of heat treatment and electron bombardment on the intensity of optically stimulated luminescence according to an embodiment.

Figure 12 illustrates the effect of an UV radiation on CR image, according to an embodiment.

5 Figure 13 illustrates the effect of an UV radiation on CR image, according to another embodiment.

Figure 14 illustrates ultraviolet enhancement of polymer emission for an embodiment.

Figure 15 illustrates ultraviolet enhancement of polymer emission for another embodiment.

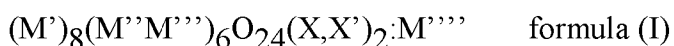
10 Figure 16 illustrates ultraviolet enhancement of polymer emission for a further embodiment.

DETAILED DESCRIPTION

In this specification, unless otherwise stated, the expression “monoatomic ion” should be understood as an ion consisting of a single atom. If an ion contains more than one atom, even if these atoms are of the same element, it is to be understood as a polyatomic ion.

15 Thus, in this specification, unless otherwise stated, the expression “monoatomic cation” should be understood as a cation consisting of a single atom.

The present description relates to a method for treating a luminescent material by electron bombardment. The material to be treated is represented by formula I



20 wherein

- M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations;

- M'' represents a trivalent monoatomic cation of an element selected from Group 13 of the IUPAC periodic table of the elements, or of a transition element selected from any of
25 Groups 3 – 12 of the IUPAC periodic table of the elements, or any combination of such cations;

- M'''' represents a monoatomic cation of an element selected from Group 14 of the IUPAC periodic table of the elements, or of an element selected from any of Groups 13 and 15 of the IUPAC periodic table of the elements, or of Zn, or any combination of such
30 cations;

- X represents an anion of an element selected from Group 17 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X is absent;
 - X' represents an anion of one or more elements selected from Group 16 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X' is absent;
 - 5 and
 - M'''' represents a dopant cation of an element selected from rare earth metals of the IUPAC periodic table of the elements, or from transition metals of the IUPAC periodic table of the elements, or of Ba, Sr, Tl, Pb, or Bi, or any combination of such cations, or wherein M'''' is absent;
 - 10 with the proviso that at least one of X and X' is present,
- the electron bombardment being carried out in a vacuum chamber having a pressure enabling formation of an electron beam.

The material of formula (I) is an optically active material that is configured to be able to retain radiation such as X-radiation exposed thereon, i.e. the material is able to trap therein
15 the radiation that it is exposed to, and to release the retained radiation as visible light when being subjected to optical stimulation, i.e. the retained radiation may be released from the material later at a desired point of time.

The present description thus concerns the materials represented by formula (I), which are electron bombarded after their manufacture. As will be demonstrated below in the
20 Experimental part, the present electron bombardment reduces the OSL afterglow of the materials, and increases the OSL signal intensity.

Electron bombardment is a generally known ionization method in which electrons interact with solid or gas phase atoms or molecules to produce ions. Ionization by electron bombardment uses highly energetic electrons to produce ions, which leads to extensive
25 fragmentation. Electron bombardment is non-selective ionization method which provides high ionization efficiency and sensitivity.

The material is preferably treated with the electron bombardment in powder form. Typically, the powder sample is spread as a thin surface onto a sample holder which is then put into a vacuum. In the chamber, the sample is being bombarded, typically small area at
30 a time until the whole sample surface has been treated. The apparatuses for carrying out electron bombardment are known as such.

The electron bombardment of material having the formula (I) is carried out in a vacuum chamber having a pressure enabling formation of an electron beam. For some typical equipment, this means a pressure of at most 15 Pa. According to another embodiment, the vacuum chamber has a pressure of at most 10 Pa, for example at most 5 Pa. The vacuum chamber can for example have a pressure of 1, 3, 5, 7, 10 or 12 Pa up to 3, 5, 7, 10, 12 or 15 Pa.

According to one embodiment, the electron bombardment is carried out using a voltage of 0.1-50 kV. For example, the voltage can be from 0.1, 1, 5, 8, 10, 12, 15, 18, 20, 22, 25, 28, 30, 32, 35, 37, 40, 42, 45 or 47 kV up to 1, 5, 8, 10, 12, 15, 18, 20, 22, 25, 28, 30, 32, 35, 37, 40, 42, 45, 47 or 50 kV.

According to another embodiment, the electron bombardment is carried out using a current of 0.01-0.5 mA. For example, the current can be from 0.01, 0.05, 0.1, 0.15, 0.2, 0.25, 0.3, 0.35, 0.4 or 0.45 mA up to 0.05, 0.1, 0.15, 0.2, 0.25, 0.3, 0.35, 0.4, 0.45 or 0.5 mA.

The electron bombardment is carried out for a period of time. The time used for electron bombardment is typically selected based on the other process parameters. According to one embodiment, the material is exposed to the electron bombardment for a time period of 1 to 40 minutes. In other embodiments, the time could be even less than one minute or more than 40 minutes, such as 45 minutes or an hour. For example, the time can be from 1, 2, 3, 4, 6, 9, 10, 15, 20, 25 or 30 minutes up to 2, 3, 4, 6, 9, 10, 15, 20, 25, 30, 35 or 40 minutes.

In one embodiment, M' represents a monoatomic cation of an alkali metal selected from a group consisting of Na, Li, K, Rb, Cs, and Fr, or any combination of such cations. In another embodiment, M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations, with the proviso that M' does not represent the monoatomic cation of Na alone. According to an embodiment, M' represents a combination of at least two monoatomic cations of different alkali metals selected from a group consisting of Li, Na, K, Rb, Cs, and Fr.

In one embodiment, M' represents a combination of at least two monoatomic cations of different alkali metals selected from Group 1 of the IUPAC periodic table of the elements. In one embodiment, M' represents a combination of at least two monoatomic cations of different alkali metals selected from Group 1 of the IUPAC periodic table of the elements, wherein the combination comprises at most 98 mol-%, at most 95 mol-%, at most 90 mol-

%, at most 85 mol-%, at most 80 mol-%, at most 70 mol-%, at most 60 mol-%, at most 50 mol-%, at most 40 mol-% of the monoatomic cation of Na, or at most 30 mol-% of the monoatomic cation of Na, or at most 20 mol-% of the monoatomic cation of Na.

In a yet further embodiment, M' represents a monoatomic cation of Li. In one embodiment, M' represents a monoatomic cation of K. In one embodiment, M' represents a monoatomic cation of Rb. In one embodiment, M' represents a monoatomic cation of Cs. In one embodiment, M' represents a monoatomic cation of Fr.

In one embodiment, M'' represents a trivalent monoatomic cation of a metal selected from a group consisting of Al and Ga, or a combination of such cations. In one embodiment, M'' represents a trivalent monoatomic cation of B. In one embodiment, M'' represents a trivalent monoatomic cation of a transition element selected from Period 4 of the IUPAC periodic table of the elements, or any combination of such cations. In one embodiment, M'' represents a trivalent monoatomic cation of an element selected from a group consisting of Cr, Mn, Fe, Co, Ni, and Zn, or any combination of such cations.

In one embodiment, M''' represents a monoatomic cation of an element selected from a group consisting of Si, Ge, Al, Ga, N, P, and As, or any combination of such cations. In one embodiment, M''' represents a monoatomic cation of an element selected from a group consisting of Si and Ge, or a combination of such cations. In one embodiment, M''' represents a monoatomic cation of an element selected from a group consisting of Al, Ga, N, P, and As, or any combination of such cations. In one embodiment, M''' represents a monoatomic cation of an element selected from a group consisting of Al and Ga, or a combination of such cations. In one embodiment, M''' represents a monoatomic cation of an element selected from a group consisting of N, P, and As, or any combination of such cations. In one embodiment, M''' represents a monoatomic cation of Zn.

In one embodiment, X represents an anion of an element selected from a group consisting of F, Cl, Br, I, and At, or any combination of such anions. In one embodiment, X represents an anion of an element selected from a group consisting of F, Cl, Br, and I, or any combination of such anions. In one embodiment, X is absent.

In one embodiment, X' represents an anion of an element selected from a group consisting of O, S, Se, and Te, or any combination of such anions. In one embodiment, X' represents an anion of one or more elements selected from a group consisting of O, S, Se, and Te, or

any combination of such anions. In one embodiment, X' represents a monoatomic or a polyatomic anion of one or more elements selected from a group consisting of O, S, Se, and Te, or any combination of such anions. In one embodiment, X' represents an anion of S. In an embodiment, X' is $(\text{SO}_4)^{2-}$. In yet another embodiment X' is absent.

- 5 The proviso that at least one of X and X' is present should in this specification, unless otherwise stated, be understood such that either X or X' is present, or such that both X and X' are present.

In one embodiment, the material is doped with at least one transition metal ion.

In one embodiment, the material is represented by formula (I), wherein M'''' represents a
10 cation of an element selected from transition metals of the IUPAC periodic table of the elements, or of Ba, Sr, Tl, Pb, or Bi, or any combination of such cations. In one embodiment, M'''' represents a cation of an element selected from transition metals of the f-block of the IUPAC periodic table of the elements. In one embodiment, M'''' represents a cation of an element selected from transition metals of the d-block of the IUPAC periodic
15 table of the elements. In one embodiment, M'''' represents a cation of an element selected from a group consisting of Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Ag, W, and Zn, or any combination of such cations. In one embodiment, M'''' represents a cation of Ti. In one embodiment, M'''' represents a dopant cation of an element selected from rare earth metals of the IUPAC periodic table of the elements. In one embodiment, M'''' represents a
20 cation of an element selected from a group consisting of Yb, Er, Tb, and Eu, or any combination of such cations. In one embodiment, M'''' represents a combination of two or more dopant cations.

In one embodiment, the material is represented by formula (I), wherein M'''' is absent. In this embodiment, the material is not doped. In one embodiment, the material represented
25 by the formula (I) comprises M'''' in an amount of 0.001 – 10 mol-%, or 0.001 - 5 mol-%, or 0.1 – 5 mol-% based on the total amount of the material.

According to a further embodiment, the material represented by formula (I) comprises residuals. These residuals originate from the manufacturing process of the material, and may be present in an amount of up to 1 mol-%, or even more, such as up to 10 mol-%.

In one embodiment, the material represented by formula (I) is selected from a group consisting of:

- (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Ga)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Cr)₆Si₆O₂₄(Cl,S)₂:Ti,
 5 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Mn)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Fe)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Co)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Ni)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Cu)₆Si₆O₂₄(Cl,S)₂:Ti,
 10 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,B)₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Mn₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Cr₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Fe₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Co₆Si₆O₂₄(Cl,S)₂:Ti,
 15 (Li_xNa_{1-x-y-z}K_yRb_z)₈Ni₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Cu₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈B₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Ga₆Si₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆Si₆O₂₄(Cl,S)₂:Ti,
 20 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Si,Zn)₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Si,Ge)₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆Zn₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆Ge₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Ga,Si,N)₆O₂₄(Cl,S)₂:Ti,
 25 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Ga,Si,As)₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Ga,N)₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈Al₆(Ga,As)₆O₂₄(Cl,S)₂:Ti,
 (Li_xNa_{1-x-y-z}K_yRb_z)₈(Al,Ga)₆Ge₆O₂₄(Cl,S)₂:Ti,

- $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cr})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Mn})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Fe})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Co})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
5 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Ni})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cu})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{B})_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Mn}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Cr}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
10 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Fe}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Co}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Ni}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Cu}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{B}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$, and
15 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Ga}_6\text{Ge}_6\text{O}_{24}(\text{Cl},\text{S})_2:\text{Ti}$,
 wherein
 $x + y + z \leq 1$, and
 $x \geq 0, y \geq 0, z \geq 0$.

Some further suitable materials are $\text{LiNa}_7\text{Al}_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$, where the amount of Sr
 20 varies from 3 to 6 mol-%. The material may also comprise Cu, for example in the amount
 of 1 mol-%. Some suitable materials represented by formula (I) can be selected from a
 group consisting of:

- $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Ga})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cr})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 25 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Mn})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Fe})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Co})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Ni})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cu})_6\text{Si}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr}$,

- $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Al}_6(\text{Ga},\text{Si},\text{As})_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Al}_6(\text{Ga},\text{N})_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Al}_6(\text{Ga},\text{As})_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Ga})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
5 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cr})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Mn})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Fe})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Co})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Ni})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
10 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{Cu})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8(\text{Al},\text{B})_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Mn}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Cr}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Fe}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
15 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Co}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Ni}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Cu}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{B}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$ and
 $(\text{Li}_x\text{Na}_{1-x-y-z}\text{K}_y\text{Rb}_z)_8\text{Ga}_6\text{Ge}_6\text{O}_{24}(\text{Br},\text{S})_2:\text{Sr},\text{Cu},$

20 wherein

$$x + y + z \leq 1, \text{ and}$$

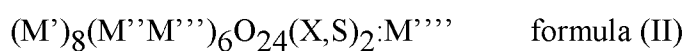
$$x \geq 0, y \geq 0, z \geq 0.$$

The material may be synthesized by a reaction according to Norrbo *et al.* (Norrbo, I.;
 Głuchowski, P.; Paturi, P.; Sinkkonen, J.; Lastusaari, M., Persistent Luminescence of
 25 Tenebrescent $\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}(\text{Cl},\text{S})_2$: Multifunctional Optical Markers. *Inorg. Chem.* 2015,
 54, 7717-7724), which reference is based on Armstrong & Weller (Armstrong, J.A.;
 Weller, J.A. Structural Observation of Photochromism. *Chem. Commun.* 2006, 1094-
 1096). As an example, stoichiometric amounts of Zeolite A and Na_2SO_4 as well as LiCl,
 NaCl, KCl and/or RbCl can be used as the starting materials. The at least one dopant may

be added as an oxide, such as TiO₂, a chloride, a sulfide, a bromide, or a nitrate. The material can be prepared as follows: Zeolite A may first be dried at 500 °C for 1 h. The initial mixture may then be heated at 850 °C in air for e.g. 2 h, 5 h, 12 h, 24 h, 36 h, 48 h, or 72 h. The product may then be freely cooled down to room temperature and ground.
 5 Finally, the product may be re-heated at 850 °C for 2 h under a flowing 12 % H₂ + 88 % N₂ atmosphere. If needed, the as-prepared materials may be washed with water to remove any excess LiCl/NaCl/KCl/RbCl impurities. The purity can be verified with an X-ray powder diffraction measurement.

The material is prepared in powder form, and is typically also used in powder form. The
 10 particle size of the powder is typically about 5-10 μm², as measured by transmission electron microscopy, the area is determined from the pictures with a watershed segmentation algorithm in the ImageJ program.

According to an alternative embodiment, the material treated has formula (II)



15 wherein

- M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations;
- M'' represents a trivalent monoatomic cation of an element selected from Group 13 of the IUPAC periodic table of the elements, or of a transition element selected from any of
 20 Groups 3 - 12 of the IUPAC periodic table of the elements, or any combination of such cations;
- M''' represents a monoatomic cation of an element selected from Group 14 of the IUPAC periodic table of the elements, or any combination of such cations;
- X represents an anion of an element selected from Group 16 of the IUPAC periodic table
 25 of the elements, or from Group 17 of the IUPAC periodic table of the elements, or any combination of such anions; and
- M'''' represents a dopant cation of an element selected from rare earth metals of the IUPAC periodic table of the elements, or from transition metals of the IUPAC periodic table of the elements, or any combination of such cations, or wherein M'''' is absent.

The various embodiments and options for the different constituents of the material according to formula (I) listed above apply *mutatis mutandis* to materials according to formula (II).

5 The material of the present description is typically non-toxic and non-expensive and also has the benefit of being reusable and recyclable.

The present description also relates to the present method of electron bombardment, followed by treating the electron bombarded material with heat treatment. According to yet another embodiment, the present electron bombardment can be carried out on a material that has been heat treated as explained below. Typically, the manufacturing process of
10 these materials includes re-heating (as will be explained below), and therefore the present heat treatment is a further re-heating of the material. As will be demonstrated below in the Experimental part, the present heat treatment reduces the OSL afterglow of the materials, and increases the OSL signal intensity.

The heat treatment is carried out for a period of time. It is presently believed that as long as
15 the temperature is not too high, there is no clear upper limit for the period of time useful for heating, but that after a certain period of time, further heating time does not have a noticeable effect on the material. The material is heated for a period of time of 15-600 minutes, such as for example from 15, 20, 25, 30, 45, 60, 90, 120, 150, 180, 210, 240, 270, 300, 330, 360, 390, 420, 450, 480, 510 or 540 minutes up to 30, 45, 60, 90, 120, 150, 180,
20 210, 240, 270, 300, 330, 360, 390, 420, 450, 480, 510, 540, 570 or 600 minutes.

The temperature used for the heat treatment is 250-900 °C, such as for example from 250,
260, 270, 280, 290, 300, 310, 320, 330, 340, 350, 360, 370, 380, 390, 400, 420, 440, 460,
480, 500, 520, 540, 560, 580, 600, 620, 640, 660, 680, 700, 720, 740, 760, 780, 800, 810,
820, 830, 840, 850, 860 or 870 °C, up to 280, 290, 300, 310, 320, 330, 340, 350, 360, 370,
25 380, 390, 400, 420, 440, 460, 480, 500, 520, 540, 560, 580, 600, 620, 640, 660, 680, 700,
720, 740, 760, 780, 800, 810, 820, 830, 840, 850, 860, 870, 880, 890 or 900 °C.

According to an embodiment, the heat treatment is carried out at a temperature of 400-600 °C, for example for 45-75 minutes. It is believed that this temperature range is especially suitable for increasing OSL intensity of at least certain materials according to formula (I).
30 According to another embodiment, the heat treatment is carried out at a temperature of 400-700 °C, for example for 45-75 minutes. It is believed that this temperature range is

especially suitable for decreasing the normal afterglow of at least certain materials according to formula (I). According to yet another embodiment, the upper temperature limit of the heat treatment is 850 °C.

5 The heat treatment is typically carried out in an oven. The material may be either gradually heated together with the oven, or it may be placed from room temperature to a ready-heated oven. In another embodiment, the material may pass through a series of ovens, having increasing temperatures. For example, the temperature difference between two consecutive ovens may be 50 or 100 °C. Also other types of devices can be used, such as chambers, furnaces etc.

10 According to an embodiment, the heat treatment is followed by cooling of the material in a step-wise manner. The material may for example be allowed to cool together with the oven, meaning that the cooling is gradual, i.e. the steps are small. The material may also be cooled by passing it through a series of ovens having decreasing temperatures. For example, the temperature difference between two consecutive ovens may be 50 or 100 °C.
15 The cooling may also be carried out by removing the material from the hot oven to room temperature.

According to an embodiment, the heat treatment is carried out in an atmosphere of air, nitrogen, argon, oxygen or mixtures thereof. A reducing gas may also be used during the heat treatment.

20 According to another embodiment, the electron bombarded material or the electron bombarded and heat treated material is further arranged in a polymer matrix. The thus obtained product is thereafter treated with ultraviolet (UV) irradiation.

It has been surprisingly found that ultraviolet irradiation treatment can be used to further improve optical properties of the material of formula (I). Ultraviolet irradiation treatment
25 enables increasing luminescence and optically stimulated luminescence intensity in computed radiography (CR) imaging, in digital radiography (DR) imaging and computed tomography (CT) imaging. The imaging techniques may use plates or detectors, or a combination of plates and detectors. The detectors may be for example X-ray detectors. In order to obtain the beneficial effect of ultraviolet irradiation, the material of formula (I) is
30 believed to require being arranged in a polymer matrix.

Without wishing to be bound by a theory, it seems that the polymer matrix acts as an energy converter that absorbs ultraviolet emission from the material, converts it to such wavelengths that can be read by CR plate reader and emits it to the reader. Thus, ultraviolet treatment makes more of the optically stimulated luminescence of the material of formula (I) visible to the CR plate reader, i.e. converts especially the high-energy part of the optically stimulated luminescence into wavelengths that CR reader can see (blue light). Ultraviolet radiation enhances the blue emission of the polymer by widening the excitation band of the polymer. Thus, the CR image becomes brighter and/or higher in contrast. Thus, ultraviolet irradiation treatment of the material arranged in polymer matrix results in increased optically stimulated luminescence signal. It has been noted that the ultraviolet treatment does not seem to affect the material as such, but only the polymer matrix, especially the surface of the polymer matrix.

According to one embodiment, the material can be arranged in a polymer matrix by using tape casting, also known as knife coating or doctor blading. Tape casting is a process where a thin sheet of ceramic or metal particle suspension fluid is cast on a substrate. The fluid may contain volatile nonaqueous solvents, a dispersant, (a) binder(s) and the dry matter, i.e. the material having formula (I). The process may comprise preparing the suspension and applying it onto a surface of a substrate. The binder may create a polymer network around the dry matter particles, while the plasticizer may function as a softening agent for the binder. When combining these substances, the tape may become resistant against cracking and flaking off when bent. The dispersant may be used to de-aggregate the particles and homogenize the suspension.

Thus, according to one embodiment the material is arranged in a polymer matrix by mixing the material with the tape casting components. Any suitable and typical tape casting components can be used, as known in the art. According to one embodiment, the tape casting components comprise ethanol Aa, ethyl methyl ketone, triton X-100, benzyl butyl phthalate and polyvinyl butyral.

The polymer matrix, i.e. tape-casting polymer, can comprise one or several different polymers. Any polymer capable to act as an energy converter, i.e. capable to be excited by the optically stimulated luminescence emission, can be used. According to one embodiment, the polymer can be benzyl butyl phthalate or polyvinyl butyral or any combination thereof.

The material, tape-casted in a polymer matrix, forms an image detector that can be used in computed radiography imaging after the beneficial UV treatment.

According to one embodiment the ultraviolet irradiation is made using an irradiation with wavelength of 200 to 310 nm. The exposure time for the UV irradiation is for example 10
5 seconds to 30 minutes. The time can thus be for example from 10, 15, 30 or 45 seconds or from 1, 5, 10 or 20 minutes up to 15, 30 or 45 seconds or 1, 5, 10, 20, 25 or 30 minutes.

According to one embodiment the ultraviolet irradiation intensity used is 4 to 6 mW/cm². Thus, ultraviolet irradiation treatment can be performed for example with wavelength of 302 nm and intensity of 4.25mW/cm² or with wavelength of 254 nm and intensity of 5.92
10 mW/cm².

The present materials may be used in X-ray imaging, in computed radiography (CR) imaging, in digital radiography (DR) imaging and in computed tomography (CT) imaging. The imaging techniques may use plates or detectors, or a combination of plates and detectors. The detectors may be for example X-ray detectors. These plates and detectors
15 may comprise the present material that has been treated, or the plate or detector (also called in general image detectors) may first be formed and thereafter the material may be treated as discussed. The material according to formula (I) may be attached to a surface for example as a coating or a film. The substrate of the plate or detector may comprise or consist of glass or polymer. The substrate may comprise or consist of a glass layer or a
20 polymer layer. The substrate may comprise (a) further layer(s). The substrate may also or alternatively comprise an attachment layer, such as a printing paper, and/or a base layer, such as a cardboard layer, or any other layer(s) where desired or needed. The image detector may comprise further layers and/or components. The image detector has the added utility of enabling the use of the material represented by formula (I) as a detector material
25 for imaging purposes. The image detector has a further added utility of making use of an optically active material being non-toxic and non-expensive compared to currently used materials such as Ba(F,Cl,Br,I)₂:Eu and CsI:Ti. The image detector has still an added utility of being reusable and recyclable. Further, the image detector can be used for point-of-care analysis without the need of complicated analysis systems.

30 The present description further relates to a device, wherein the device comprises a material according to one or more embodiments described in this specification. In one embodiment,

the device is an ultraviolet radiation sensor, an ultraviolet radiation detector, or an ultraviolet radiation indicator. In one embodiment, the device is an X-radiation sensor, an X-radiation detector, an X-radiation indicator, or an X-radiation dose indicator.

5 The present method of electron bombardment of the material has the effect of decreasing afterglow of the present materials. The present description thus relates also to a method for decreasing afterglow of a material represented by formula (I) as defined above, by carrying out the electron bombardment described above. According to a preferred embodiment, the decreased afterglow is afterglow of optically stimulated luminescence. Preferably, the optical stimulation being carried out with a laser. The effect is obtained especially when
10 the material is both electron bombarded and heat treated.

In this specification, unless otherwise stated, the expressions "afterglow", "persistent luminescence", "phosphorescence", or any corresponding expression should be understood as referring to the phenomenon encountered in materials which make them glow in the dark after having been excited with ultraviolet or visible light or with higher energy radiation.

15 A further effect of the present invention is the increase of intensity of optically stimulated luminescence. Thus, the present description also relates to a method for increasing intensity of optically stimulated luminescence of a material represented by formula (I) as defined above, by carrying out the electron bombardment as described above. The effect is obtained especially when the material is both electron bombarded and heat treated.

20 A still further aspect of this description is a method for increasing contrast of a computed radiography plate, by using a material represented by formula (I) and treated with ultraviolet (UV) irradiation, as explained above.

It will be understood that the benefits and advantages described above may relate to one embodiment or may relate to several embodiments. The embodiments are not limited to
25 those that solve any or all of the stated problems or those that have any or all of the stated benefits and advantages. The embodiments described herein before may be used in any combination with each other. Several of the embodiments may be combined together to form a further embodiment of the invention. An image detector, or a use, to which the current specification is related, may comprise at least one of the embodiments described
30 hereinbefore.

In the following Experimental part, Examples 1 to 3 are given to further illustrate the invention and Figures 1 to 10 are described.

EXPERIMENTAL PART

Example 1 - Preparation of the materials

5 The materials tested had the formula $\text{LiNa}_7\text{Al}_6\text{Si}_6\text{O}_{24}(\text{Br,S})_2\text{:Sr}$, with various amounts of Sr. One sample also had some Cu. The amounts of Sr (and Cu) are given below in Table 1.

During the synthesis of the materials, zeolite A was first dried at 500 °C for 1 h. Stoichiometric amounts of zeolite A, NaBr, Na_2SO_4 and LiBr were used as the starting materials. The dopants were added as bromides (SrBr_2 , CuBr when used). The following
10 conditions were used: first heating was carried out at 800 °C in air for 5 hours, then a second heating at 850 °C under an atmosphere of 12 % H_2 and 88 % N_2 for 2 hours. For the material MT84, the first heating was done for 5 hours in 850 °C, removed from the oven at 850 °C and thus cooled down quickly. The second heating was as above.

Thereafter, the material was electron bombarded as described below, and thereafter tested
15 as also described below. For each material, the MTxxa is the reference material that was not electron bombarded, the MTxxb etc. is the electron bombarded sample. These references a-g may not be used in the explanation of the results.

Sample	Amount of Sr, other components	Electron bombardment time/surface area
MT55a	6 mol-% Sr 1 mol-% Cu	Reference, not electron bombarded
MT55b	6 mol-% Sr 1 mol-% Cu	Electron bombarded
MT57a	6 mol-% Sr	Reference, not electron bombarded
MT57b	6 mol-% Sr	Electron bombarded
MT80a	6 mol-% Sr	Reference, not electron bombarded
MT80b	6 mol-% Sr	Electron bombarded
MT82a	6 mol-% Sr	Reference, heat treated at 600 °C but not electron bombarded
MT82b	6 mol-% Sr	Heat treated at 600 °C, then electron bombarded
MT83a	6 mol-% Sr	Reference, not electron bombarded
MT83b	6 mol-% Sr	Electron bombarded for 30 seconds
MT83c	6 mol-% Sr	Electron bombarded for 1 minute
MT83d	6 mol-% Sr	Electron bombarded for 5 minutes
MT83e	6 mol-% Sr	Electron bombarded for 10 minutes
MT83f	6 mol-% Sr	Electron bombarded for 20 minutes
MT83g	6 mol-% Sr	Electron bombarded for 30 minutes
MT84a	6 mol-% Sr	Reference, not electron bombarded
MT84b	6 mol-% Sr	Electron bombarded
MT85a	6 mol-% Sr	Reference, not electron bombarded
MT85b	6 mol-% Sr	Electron bombarded

Table 1

Example 2 - Electron bombardment treatment

The temperature and pressure in the laboratory was kept at 20 °C and 1 atm.

5 Approximately 30 mg of sample powder was spread as a thin surface onto a glass sample holder which was then put into the vacuum chamber Nuclide Corp. ELM2B connected to a Franklin Electric model 1102685400 vacuum pump providing continuous suction. The control unit Nuclide Corp. ELM2EX was then used to control the voltage and current levels for the electron bombardment. The voltage was kept at 5 kV and the current was

10 kept at 1.0 mA for samples other than MT82 and MT83, the conditions for MT82 and MT83 being given below in Table 2. When the equipment's pressure reading was below 13 Pa, the electron beam was visible and could be visually adjusted with magnets that were

held on both sides of the vacuum chamber's window. In these experiments, the visible beam was kept oval shaped, measuring 7 mm x 30 mm. This area of the sample was bombarded for 30 min (other samples than MT82b and MT83), after which a new, unexposed area was bombarded for the same time until the whole surface of the sample surface had been exposed to a same current, voltage and time product. For the samples 5 MT83b-MT83g, the time of electron bombardment of each portion was as indicated in Table 1, while the voltage and current as well as the measured pressure are given below in Table 2. The samples MT82a and MT82b had been heat treated before electron bombardment.

Sample	Time/surface area	Pressure	Voltage	Current
MT82b	20 minutes	6.13-6.53 Pa	8.75-10.0 kV	0.74-0,80 mA
MT83b	30 seconds	9.33 Pa	5.50 kV	0.80 mA
MT83c	1 minute	8.67-9.07 Pa	6-6.25 kV	0.80 mA
MT83d	5 minutes	7.33-8.40Pa	6.5-6.8 kV	0.80 mA
MT83e	10 minutes	6.80-9.07 Pa	6.75-13.5 kV	0.80 mA
MT83f	20 minutes	6.00-6.67 Pa	14.0-18.0 kV	0.80 mA
MT83g	30 minutes	7.73-8.13 Pa	7.0 kV	0.80 mA

10 Table 2

Example 3 - UV irradiation treatment

The materials used were as indicated in Table 3.

Sample	Amount of Sr, other components
MT55a	6 mol-% Sr 1 mol-% Cu
MT55b	6 mol-% Sr 1 mol-% Cu
MT57a	6 mol-% Sr
MT57b	6 mol-% Sr

Table 3

Example 3a

The materials MT55 and MT57 were each suspended separately in a tape-cast polymer matrix comprising ethanol Aa (15 weight-%), ethyl methyl ketone (= 2-butanone, 30 weight-%), 2-[4-(2,4,4-trimethylpentan-2-yl)phenoxy]ethanol (sold under tradename
5 Triton X-100, 2 weight-%), benzyl butyl phthalate (BBP, 6 weight-%) and polyvinyl butyral (PVB, 7 weight-%), the amount of MT55 or MT57 being 40 weight-%. The mixture was then cast onto a polyester projector transparency, and subjected to UV irradiation at 20 °C and normal air pressure (1 atm). A 302 nm handheld UVP model UVM-57, 6 W lamp was placed directly on top of the sample and switched on, exposing
10 the sample to UV radiation for 5-30 min with an irradiance of 4.25 mW/cm². When the photochromism of the material had faded, the enhanced glow of the polymer was visible under 302 nm and 365 nm UV light.

Example 3b

The material of formula Na₈Al₆Si₆O₂₄(Cl,S)₂ was suspended in the same polymer matrix
15 as in Example 3a and subjected to 254 nm UV irradiation from a handheld UVP model UVLS-24 EL, 4 W 254/365 nm lamp, also at 20 °C and normal air pressure (1 atm). The lamp was placed directly on top of the sample and switched on, exposing the sample to UV radiation for between 10 seconds and 30 minutes with an irradiance of 5.92 mW/cm². When the photochromism of the material had faded, the enhanced glow of the polymer was
20 visible under 302 nm and 365 nm UV light.

Example 3c

The tape-cast polymer as well as the polyester projector transparency of Example 3a (i.e. without any material according to formula (I)) were exposed to UV irradiation by the same
25 302 nm lamp for 25 minutes at an irradiance of 4.25 mW/cm². After irradiation ceased, the enhanced glow of the polymers was visible immediately under 302 nm and 365 nm UV light.

Example 4 – Heat treatment

The samples MT82a and MT82b were heat treated as described below. The below explained heat-treatment was thus a third heating of the material, since the beginning of its manufacturing.

- 5 A small amount of each material (approximately 5-10 mg) was placed into a small thermogravimetric analysis crucible. Each sample was then heated in a Nabertherm N3/C8 muffle furnace at the default heating rate of approximately 20 °C/min up to a temperature of 600 °C in air at normal pressure (1 atm). The sample was kept for 1 h at the desired temperature before being allowed to cool freely inside the furnace. Once the sample had
- 10 cooled down, it was collected and its optically stimulated luminescence (OSL) was measured (as explained in more detail below).

MT82a is the reference material that was not heat-treated, while the MT82b is the heat-treated sample. After heat-treatment, the samples were submitted to electron bombardment as described above.

15 Optically stimulated luminescence (OSL) measurement

- OSL intensity and afterglow were measured as follows. The temperature and pressure in the laboratory were kept at 20 °C and 1 atm. First, the sample powder was put into a glass capillary tube measuring 75 x 2.0 mm (Marienfeld no. 29 402 02). The sample was then irradiated with a Hg discharge lamp providing 254 nm emission and put at a distance
- 20 where the irradiance was 0.12 mW/cm². The sample tube was spun at a rate of 70 revolutions per second and is irradiated for 60 s while spinning. The sample tube was then put into the middle of a measuring chamber with a 44.4 mm inner diameter and kept there for 12 hours (MT57, MT80, MT85, MT80, MT84 and MT85 in Figures 1, 2, 3, 6, 7 and 8, respectively), for 24 hours (MT83 in Figures 9 and 10) or for 5 hours (MT82 in Figure 11)
- 25 to eliminate the initial afterglow. The OSL intensity was measured by irradiating the sample with a 635 nm laser supplying 96 mA power output for 0.1 s. One measurement set was an average of 100 measurements with 1.0 s scan time per measurement or an average of 10 measurements with 10.0 s scan time per measurement using a sampling step of 1 ms or 10 ms, respectively (10 measurements with 10.0 s scan time per measurement using 10
- 30 ms sampling step for MT57, MT80, MT85, MT80, MT84 and MT85 in Figures 1, 2, 3, 6, 7

and 8, respectively; 100 measurements with 1.0 s scan time per measurement using 1 ms sampling step for MT83 and MT82 in Figures 9, 10 and 11).

The adjustment of the laser beam was carried out with a 600 nm long wave pass filter, and the emission side consists of Thorlabs LA1951-B F25.4 mm lens, short wave pass filter
 5 550 nm, short wave pass filter 600 nm, Thorlabs LA1951-B F25.4 mm lens, and finally a photomultiplier tube, respectively. The photomultiplier tube was connected to a Stanford Research Systems SR570 low noise current preamplifier (parameters: bias voltage POS, filter type: none, input offset: 1 pA, gain mode: low noise, sensitivity: 50 nA/V; (20 nA/V used for non-treated and 50 nA/V for treated MT57, MT80, MT85, MT80, MT84 and
 10 MT85 in Figures 1, 2, 3, 6, 7 and 8, respectively; 50 nA/V used for MT83 and MT82 in Figures 9, 10 and 11) and to a Stanford Research Systems PS325 high voltage power supply unit. The preamplifier was connected to ports 7 and 8 in a National Instruments NI USB-6251 multifunction I/O device. A wire loop from ports 4 and 5 to ports 15 and 16 was connected in parallel to a Tektronic TDS 420 oscilloscope and a Newport laser diode
 15 driver model 5060. The laser diode driver was connected to a Thorlabs LDM9LD laser diode, which was connected to a TE Technology TL-48-20 temperature control unit. The temperature of the laser diode was kept at 18.0 °C and controlled with a Wentronic DF17132SB DL PSV laboratory power supply set to 2.8 V.

Normal afterglow measurement

20 The normal afterglow was measured with a luminance spectrometer setup: the sample was irradiated for 5 minutes under 254 nm UV light with UVC irradiance of 3.4 mW/cm² hitting the sample, after which the afterglow measurement was started immediately with a Hagner Evacuation Route Photometer ERP-105 coupled to a Hagner SD 27 detector. The signal coming from the instrument was converted to volts with a Fluke multimeter.
 25 FlukeView Forms PC application reads the value of the multimeter every second, and the value with a certain multiplication factor (depending on the sensitivity setting on the ERP-105) is used to calculate the actual luminance in mcd/m².

Results

30 Figures 1 to 3 illustrate the decrease of optically stimulated luminescence afterglow in electron bombarded material compared to non-treated material. In these Figures, x-axis stands for “time/s”, whereas y-axis stands for “normalized intensity”. Solid line and dashed

line describe electron bombarded material and non-treated material correspondingly. The material tested was MT57 in Figure 1, MT80 in Figure 2 and MT85 in Figure 3. In these Figures, as the results are normalised, only the shape of the curve is significant.

Figures 4 and 5 illustrate the decrease of normal afterglow in electron bombarded material compared to non-treated material. In these figures, x-axis stands for “time” in seconds, whereas y-axis stands for “luminance” in mcd/m^2 . The lower line represents the electron bombarded material, whereas the upper line represents the non-treated material. The material tested was MT55 in Figure 4 and MT57 in Figure 5.

From Figures 1 to 5 it can be seen that electron bombardment considerably decreases both the OSL and normal afterglow.

Figures 6-8 illustrate the increase of intensity of optically stimulated luminescence in electron bombarded material. The tested materials were MT80 in Figure 6, MT84 in Figure 7 and MT85 in Figure 8. The Figures show the OSL intensity on the y-axis (in arbitrary units, a.u.) and time (in seconds) on the x-axis. In Figure 6, the upper curve is from the electron bombarded sample of MT80, while the lower was from the non-treated sample of the same MT80. In Figures 7 and 8, the same order of the curves was observed, although the results were closer to one another. In these Figures, the sensitivity was set higher in the reference sample (i.e. non-treated sample), thus the intensity of the electron-bombarded sample was in fact even higher with respect to the non-treated sample, than what is immediately apparent from the Figures. The difference was about 2.5 fold.

Figures 9 and 10 illustrate the effect of the time of electron bombardment on the intensity of OSL of the material MT83. Figures 9 and 10 are the same results, but Figure 10 shows the results stacked, for better visual assessment. The scale on y-axis in Figure 10 is thus not representative, only the differences in the different lines. In Figure 10, the times of bombardment from top line towards the lowest line were 30 minutes, 20 minutes, 10 minutes, 5 minutes, 1 minute, 30 seconds and no bombardment. The Figure clearly illustrates that for this particular material, the optimum electron bombardment time in order to increase the OSL intensity is 1-10 minutes. The same graphs are shown in Figure 9, where the different lines are best distinguished from one another at times 0-0.1 seconds. The line that raises the highest is for the bombardment time 10 minutes, the second highest

for 1 minute, the third highest for 5 minutes, thereafter 20 minutes, while the others are approximately the same.

Figure 11 illustrates the effect of electron bombardment and heat treatment on the intensity of optically stimulated luminescence according to an embodiment. The sample was MT82, heat-treated and then electron bombarded as explained above. Figure 11 also shows the results stacked, for better visual assessment. The scale on y-axis in Figure 11 is thus not representative, only the differences in the different lines. The upper line shows the result for the heat-treated but not electron bombarded sample MT82a, while the lower line is for the sample treated at 600 °C, followed by electron bombardment for 20 minutes (sample MT82b). As can be seen, the increase in OSL intensity is clearly higher for the sample MT82b. Figures 12 and 13 illustrate the effect of an UV radiation on CR image. The material used in Figure 12 was MT55 (Example 3a) and in Figure 13, the material was MT57 (Example 3a). In both Figures the left-most picture is the plate prior to UV treatment. The lower half of both plates was treated with one UV treatment (302 nm/20 min/4.25 mW/cm²), whereas the upper half was left untreated. After the UV treatment, X-rays (80 kV, 25 mA) were used to produce the image. The different pictures show the image of the plate after different amounts of X-ray exposures. In both Figures, the second plate was exposed to X-rays ten times (total dose 101 mGy), the third plate 8 times (total dose 80 mGy), the fourth plate 5 times (total dose 50 mGy) and the fifth plate 1 time (total dose 10 mGy), when reading from left to right.

Thus, it can be seen from Figures 12 and 13 that there is a much higher contrast, i.e. the signal is more intense, of the CR image in the region (lower half) which was subjected to UV treatment. The Figures also show that the UV treatment gives a readable CR image also at lower X-ray doses, which is not the case with a corresponding material without the UV treatment.

Figures 14 to 16 illustrate ultraviolet enhancement of polymer emission, i.e. without any material according to formula (I), obtained according to Example 3c. The tape-cast polymer as explained above was used in Figures 14 and 15, while the polyester projector transparency was used in Figure 16. In each experiment, unexposed and UV-treated tape-cast polymers were compared. All the Figures represent intensity (a.u., on the y-axis) as a function of wavelength (nm, on the x-axis). The measurements were an average of 10 measurements, with 1 second integration time.

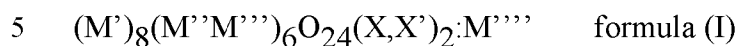
From Figure 14, it can be seen that the UV-treated polymer, which is the upper curve between wavelengths of about 450-485 nm, thus has a more intense blue emission. Figure 15, in turn, shows the excitation spectra for blue emission for unexposed (marked with squares) and UV-treated polymer (marked with spheres), wherein it can be seen that the UV-treated polymer has a broader excitation band, which is thought to be the reason for brighter blue emission.

Finally, Figure 16 shows emission spectra of unexposed and UV-treated polyester transparency. From the Figure it can be seen that two different emission bands of this polymer (420 nm and 480 nm) are both enhanced by UV treatment.

10

CLAIMS

1. A method for increasing intensity of optically simulated luminescence of a material by carrying out an electron bombardment treatment , wherein the material is represented by formula (I)



wherein

- M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations;
 - M'' represents a trivalent monoatomic cation of an element selected from Group 13 of the IUPAC periodic table of the elements, or of a transition element selected from any of Groups 3 – 12 of the IUPAC periodic table of the elements, or any combination of such cations;
 - M''' represents a monoatomic cation of an element selected from Group 14 of the IUPAC periodic table of the elements, or of an element selected from any of Groups 13 and 15 of the IUPAC periodic table of the elements, or of Zn, or any combination of such cations;
 - X represents an anion of an element selected from Group 17 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X is absent;
 - X' represents an anion of one or more elements selected from Group 16 of the IUPAC periodic table of the elements, or any combination of such anions, or wherein X' is absent;
 - and
 - M'''' represents a dopant cation of an element selected from rare earth metals of the IUPAC periodic table of the elements, or from transition metals of the IUPAC periodic table of the elements, or of Ba, Sr, Tl, Pb, or Bi, or any combination of such cations, or wherein M'''' is absent;
- with the proviso that at least one of X and X' is present,
and the electron bombardment is carried out in a vacuum chamber having a pressure enabling formation of an electron beam.

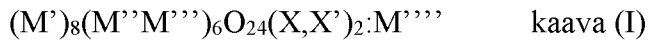
2. The method according to claim 1, wherein the electron bombardment is carried out using a voltage of 0.1-50 kV and a current of 0.01-0.5 mA.

3. The method according to claim 1 or 2, wherein the material is exposed to the electron bombardment for a time period of 1-40 min.
4. The method according to any one of the preceding claims, wherein the electron bombardment is carried out at a pressure of at most 15 Pa.
- 5 5. The method according to any one of the preceding claims, wherein M' represents a monoatomic cation of an alkali metal selected from Group 1 of the IUPAC periodic table of the elements, or any combination of such cations, with the proviso that M' does not represent the monoatomic cation of Na alone.
6. The method according to any one of the claims 1-4, wherein M' represents a
10 combination of at least two monoatomic cations of different alkali metals selected from Group 1 of the IUPAC periodic table of the elements.
7. The method according to any one of the claims 1-4, wherein M' represents a combination of at least two monoatomic cations of different alkali metals selected from a group consisting of Li, Na, K, Rb, Cs, and Fr.
- 15 8. The method according to any one of the preceding claims, wherein M'' represents a trivalent monoatomic cation of a metal selected from a group consisting of Al and Ga, or a combination of such cations.
9. The method of any one of claims 1-7, wherein M'' represents a trivalent monoatomic cation of B.
- 20 10. The method of any one of the preceding claims, wherein M''' represents a monoatomic cation of an element selected from a group consisting of Si and Ge, or a combination of such cations.
11. The method of any one of claims 1-9, wherein M''' represents a monoatomic cation of an element selected from a group consisting of Al, Ga, N, P, and As, or any combination of
25 such cations.
12. The method of any one of the preceding claims, wherein X represents an anion of an element selected from a group consisting of F, Cl, Br, I, and At, or any combination of such anions.

13. The method of any one of the preceding claims, wherein X' represents a monoatomic or a polyatomic anion of one or more elements selected from a group consisting of O, S, Se, and Te, or any combination of such anions.
14. The method of any one of the preceding claims, wherein M'''' represents a cation of an
5 element selected from a group consisting of Yb, Er, Tb, and Eu, or any combination of such cations.
15. The method of any one of claims 1-13, wherein M'''' represents a cation of an element selected from a group consisting of Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Ag, W, and Zn, or any combination of such cations.
- 10 16. The method according to any one of the preceding claims, further comprising treating the electron bombarded material with heat, at a temperature of 250-900 °C for a period of time of 15 to 600 minutes.
17. The method according to any one of the preceding claims, further comprising arranging the material in a polymer matrix and treating the material with ultraviolet irradiation, using
15 - an irradiation with a wavelength of 200-310 nm, and
- exposure time of 10 seconds to 30 min.
18. A method for decreasing afterglow of a material represented by formula (I) as defined in claim 1, by carrying out the electron bombardment treatment according to any of the claims 1-17.
- 20 19. The method according to claim 18, wherein the decreased afterglow is afterglow of optically stimulated luminescence, the optical stimulation being carried out with laser.
20. A method for increasing contrast of an image selected from computed radiography, computed tomography and digital radiography, by using a material treated with the method of claim 17.

PATENTTIVAATIMUKSET

1. Menetelmä materiaalin optisesti simuloidun luminesenssin intensiteetin lisäämiseksi toteuttamalla elektronipommituskäsittely, jossa materiaalia edustaa kaava (I)



5 jossa

- M^{\prime} tarkoittaa monoatomista kationia alkalimetallista, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 1, tai mitä tahansa tällaisten kationien yhdistelmää;

10 - $M^{\prime\prime}$ tarkoittaa trivalenttista monoatomista kationia alkuaineesta, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 13, tai siirtymäalkuaineesta, joka on valittu mistä tahansa IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmistä 3 – 12, tai mitä tahansa tällaisten kationien yhdistelmää;

15 - $M^{\prime\prime\prime}$ tarkoittaa monoatomista kationia alkuaineesta, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 14, tai alkuaineesta, joka on valittu mistä tahansa IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmistä 13 ja 15, tai Zn:stä, tai mitä tahansa tällaisten kationien yhdistelmää;

- X tarkoittaa anionia alkuaineesta, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 17, tai mitä tahansa tällaisten anionien yhdistelmää, tai X puuttuu;

20 - X^{\prime} tarkoittaa anionia yhdestä tai useammasta alkuaineesta, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 16, tai mitä tahansa tällaisten anionien yhdistelmää, tai X^{\prime} puuttuu; ja

25 - $M^{\prime\prime\prime\prime}$ tarkoittaa dopanttikationia alkuaineesta, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän harvinaisista maametalleista, tai IUPAC:n alkuaineiden jaksollisen järjestelmän siirtymämetalleista, tai Ba:sta, Sr:stä, Tl:stä, Pb:stä, tai Bi:stä, tai mitä tahansa tällaisten kationien yhdistelmää, tai $M^{\prime\prime\prime\prime}$ puuttuu;

sillä ehdolla, että vähintään toinen X :stä ja X^{\prime} :stä on läsnä,

ja elektronipommitus toteutetaan tyhjiökammiossa, jolla on paine, joka mahdollistaa elektronisuihkun muodostuksen.

30 2. Patenttivaatimuksen 1 mukainen menetelmä, jossa elektronipommitus toteutetaan käyttäen 0,1-50 kV:n jännitettä ja 0,01-0,5 mA:n sähkövirtaa.

3. Patenttivaatimuksen 1 tai 2 mukainen menetelmä, jossa materiaali altistetaan elektronipommitukselle 1-40 minuutin ajanjakson ajan.
4. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa elektronipommitus toteutetaan korkeintaan 15 Pa:n paineessa.
- 5 5. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa M' tarkoittaa monoatomista kationia alkalimetallista, joka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 1, tai mitä tahansa tällaisten kationien yhdistelmää, sillä ehdolla, että M' ei tarkoita pelkästään Na:n monoatomista kationia.
6. Jonkin patenttivaatimuksista 1-4 mukainen menetelmä, jossa M' tarkoittaa vähintään kahden monoatomisen kationin yhdistelmää eri alkalimetalleista, jotka on valittu IUPAC:n alkuaineiden jaksollisen järjestelmän ryhmästä 1.
- 10 7. Jonkin patenttivaatimuksista 1-4 mukainen menetelmä, jossa M' tarkoittaa vähintään kahden monoatomisen kationin yhdistelmää eri alkalimetalleista, jotka on valittu ryhmästä, joka koostuu Li:stä, Na:sta, K:sta, Rb:stä, Cs:stä, ja Fr:stä.
- 15 8. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa M'' tarkoittaa trivalenttista monoatomista kationia metallista, joka on valittu ryhmästä, joka koostuu Al:stä ja Ga:sta, tai tällaisten kationien yhdistelmää.
9. Jonkin patenttivaatimuksista 1-7 mukainen menetelmä, jossa M'' tarkoittaa B:n trivalenttista monoatomista kationia.
- 20 10. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa M''' tarkoittaa monoatomista kationia alkuaineesta, joka on valittu ryhmästä, joka koostuu Si:stä ja Ge:stä, tai tällaisten kationien yhdistelmää.
11. Jonkin patenttivaatimuksista 1-9 mukainen menetelmä, jossa M''' tarkoittaa monoatomista kationia alkuaineesta, joka on valittu ryhmästä, joka koostuu Al:stä, Ga:sta, N:stä, P:stä, ja As:stä, tai mitä tahansa tällaisten kationien yhdistelmää.
- 25 12. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa X tarkoittaa anionia alkuaineesta, joka on valittu ryhmästä, joka koostuu F:stä, Cl:stä, Br:stä, I:stä, ja At:stä, tai mitä tahansa tällaisten anionien yhdistelmää.

13. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa X' tarkoittaa monoatomista tai polyatomista anionia yhdestä tai useammasta alkuaineesta, joka on valittu ryhmästä, joka koostuu O:sta, S:stä, Se:stä, ja Te:stä, tai mitä tahansa tällaisten anionien yhdistelmää.
- 5 14. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, jossa M'''' tarkoittaa kationia alkuaineesta, joka on valittu ryhmästä, joka koostuu Yb:stä, Er:stä, Tb:stä, ja Eu:sta, tai mitä tahansa tällaisten kationien yhdistelmää.
15. Jonkin patenttivaatimuksista 1-13 mukainen menetelmä, jossa M'''' tarkoittaa kationia alkuaineesta, joka on valittu ryhmästä, joka koostuu Ti:stä, V:stä, Cr:stä, Mn:stä, Fe:stä,
10 Co:sta, Ni:stä, Cu:sta, Ag:stä, W:stä, ja Zn:stä, tai mitä tahansa tällaisten kationien yhdistelmää.
16. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, joka käsittää edelleen, että käsitellään elektronipommitettua materiaalia lämmöllä, 250-900 °C:n lämpötilassa 15 - 600 minuutin ajanjakson ajan.
- 15 17. Jonkin edeltävistä patenttivaatimuksista mukainen menetelmä, joka käsittää edelleen, että järjestetään materiaali polymeerimatriisiin ja käsitellään materiaalia ultravioletisäteilytyksellä käyttäen
- säteilytystä aallonpituudella, joka on 200-310 nm, ja
 - altistusaikaa, joka on 10 sekuntia - 30 min.
- 20 18. Menetelmä patenttivaatimuksessa 1 määritellyn materiaalin, jota edustaa kaava (I), jälkiloisten vähentämiseksi, toteuttamalla jonkin patenttivaatimuksista 1-17 mukainen elektronipommituskäsittely.
19. Patenttivaatimuksen 18 mukainen menetelmä, jossa vähennetty jälkiloiste on optisesti stimuloidun luminesenssin jälkiloiste, ja jossa optinen stimulointi on toteutettu laserilla.
- 25 20. Menetelmä tietokoneradiografiasta, tietokonetomografiasta ja digitaalisesta radiografiasta valitun kuvan kontrastin lisäämiseksi käyttämällä materiaalia, joka on käsitelty patenttivaatimuksen 17 mukaisella menetelmällä.

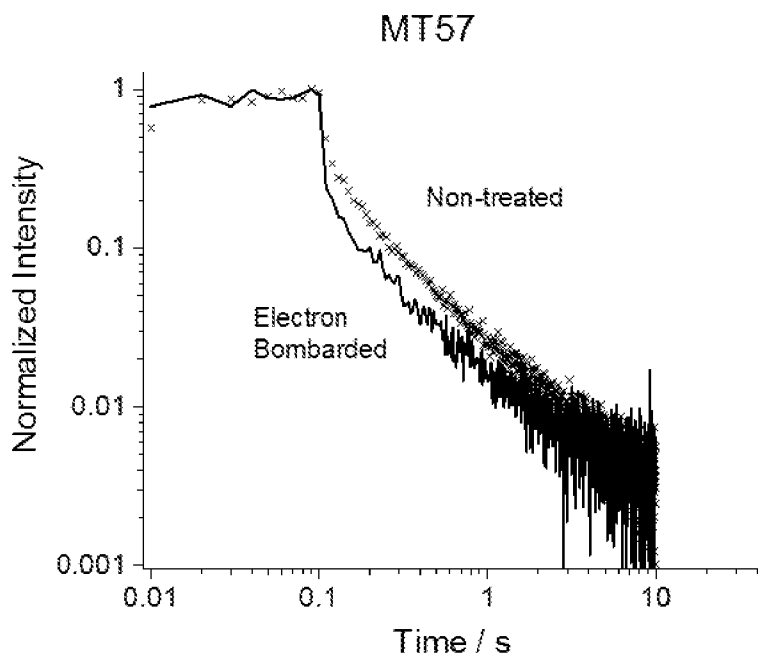


Fig. 1

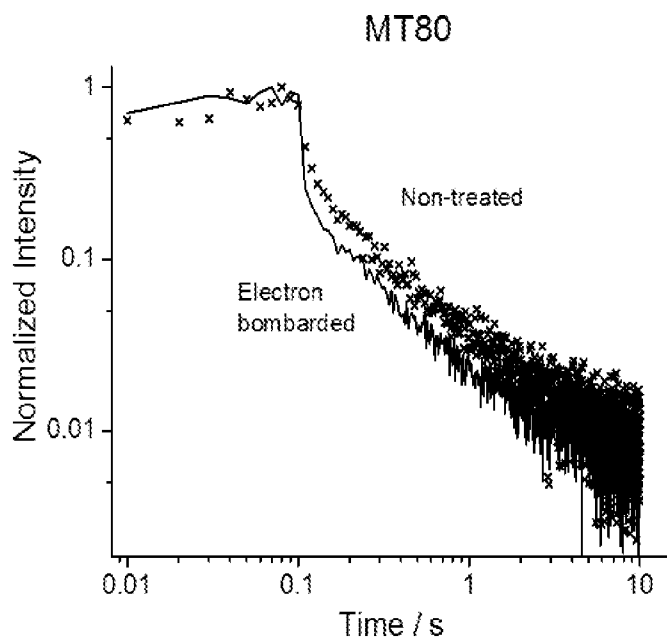


Fig. 2

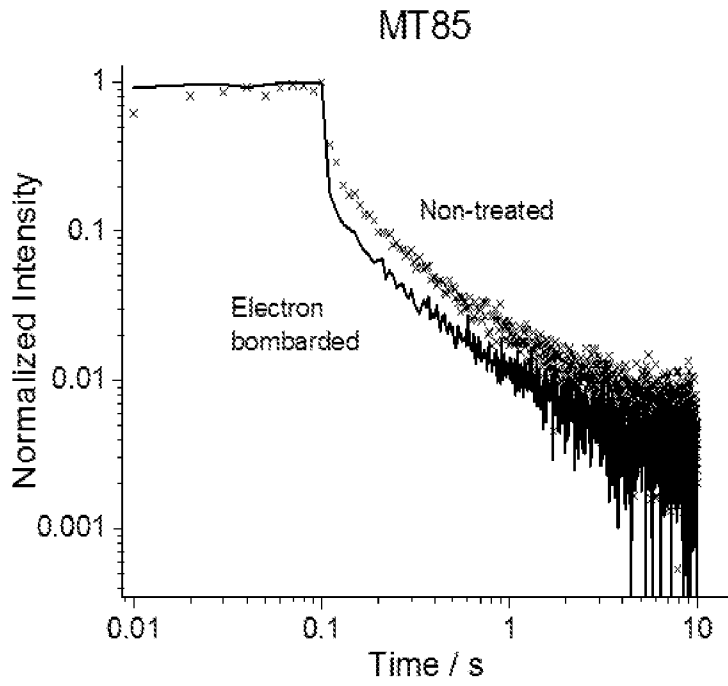


Fig. 3

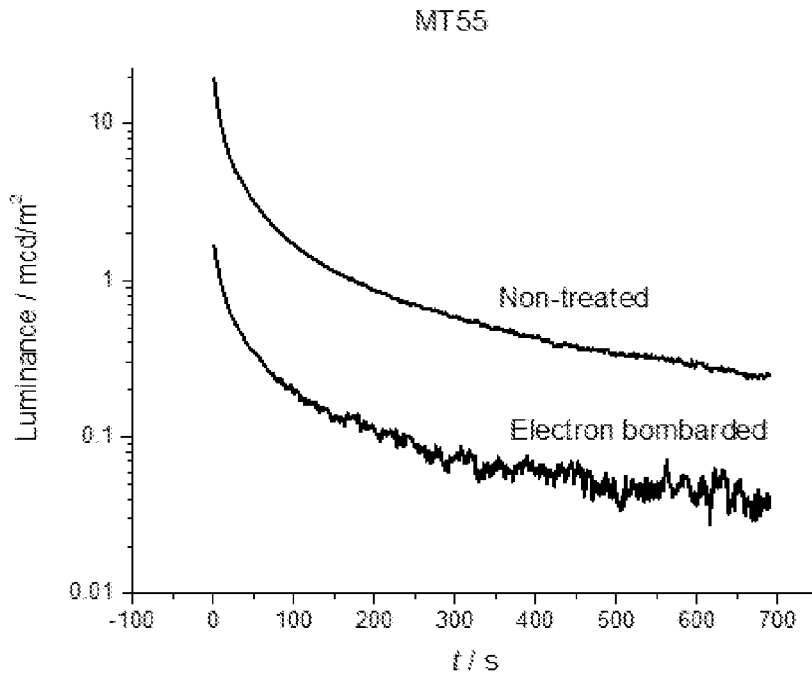


Fig. 4

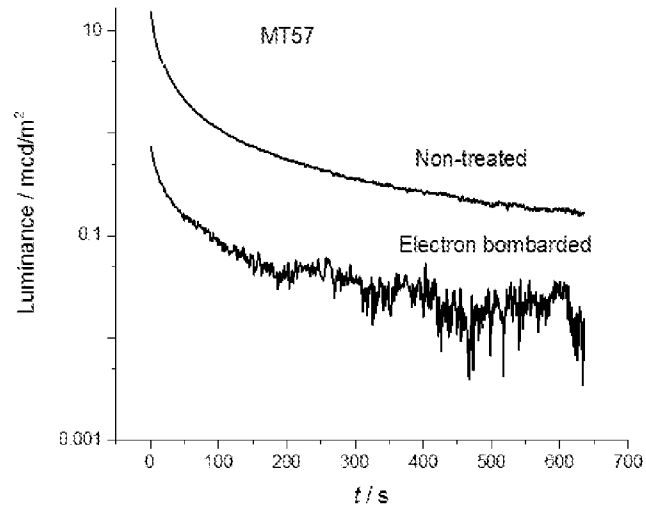


Fig. 5

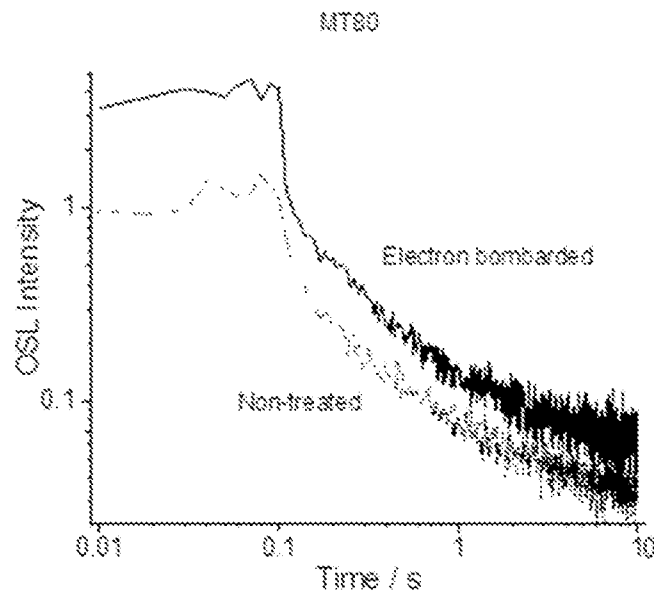


Fig. 6

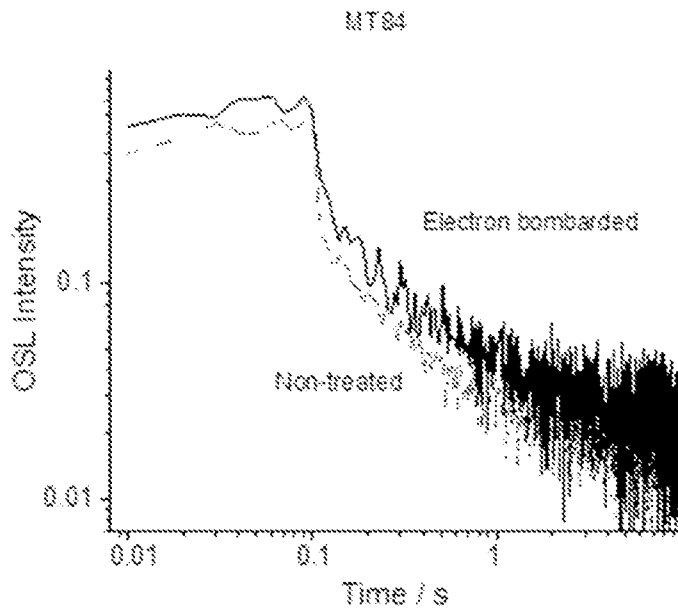


Fig. 7

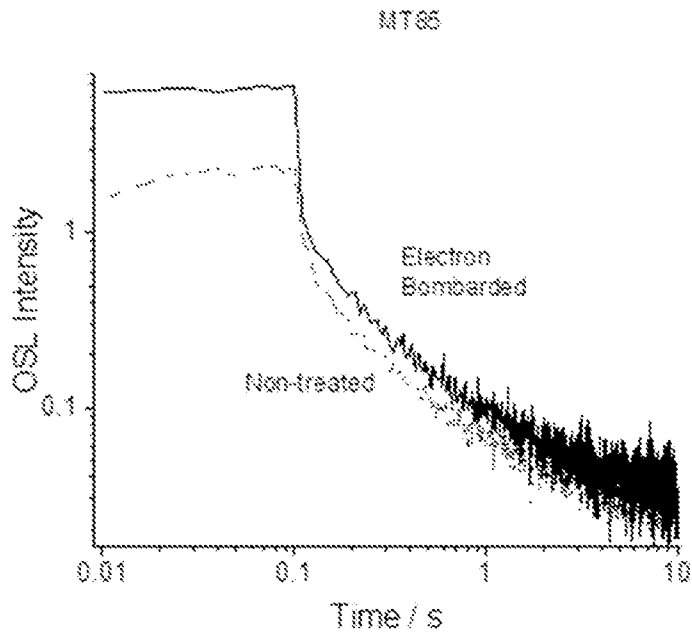


Fig. 8

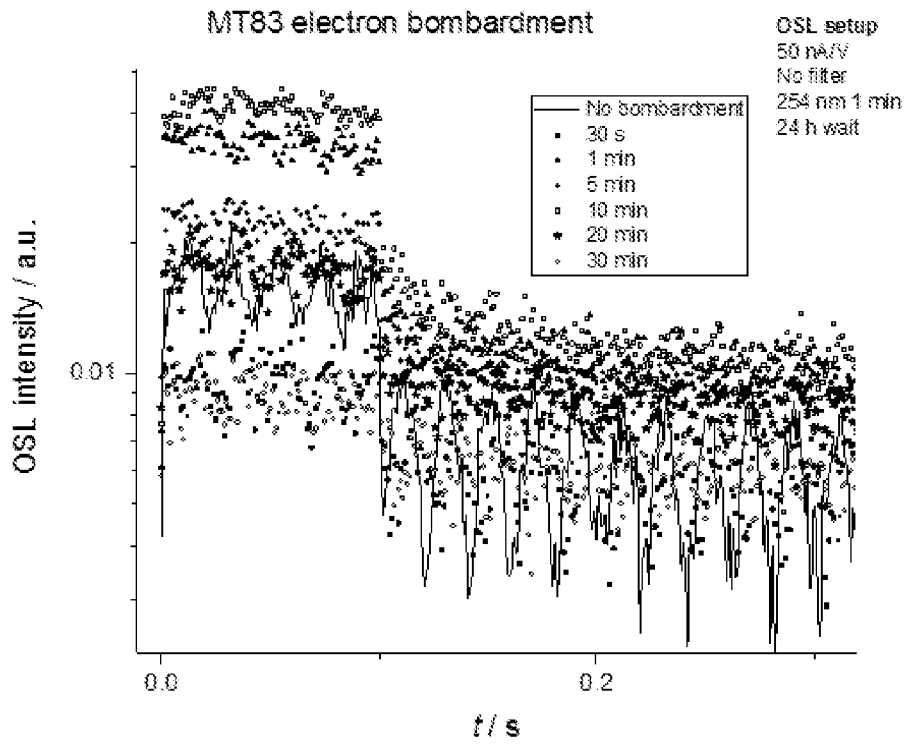


Fig. 9

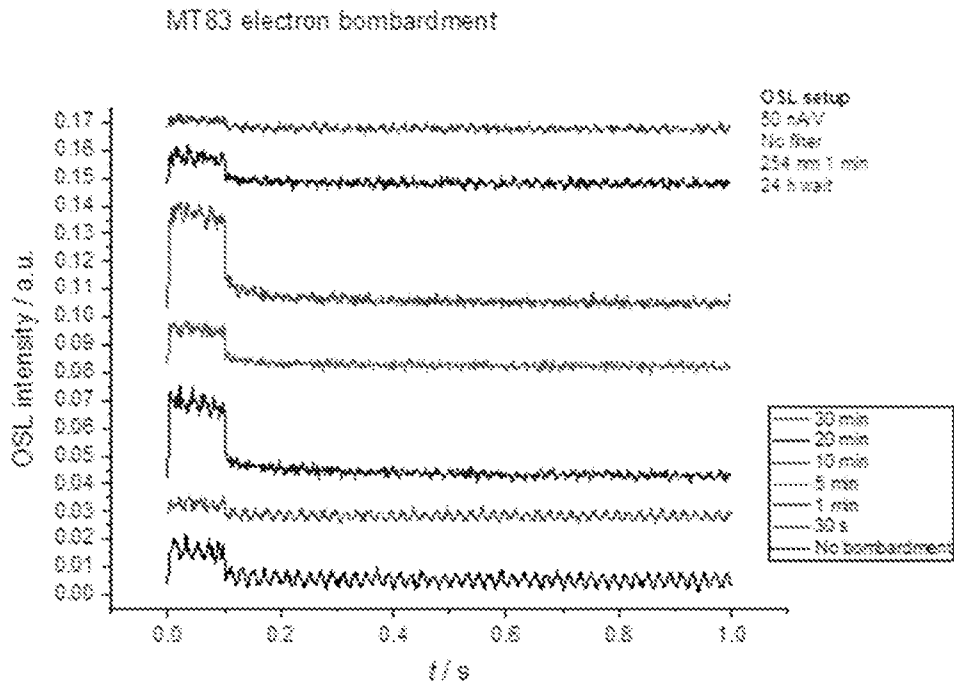


Fig. 10

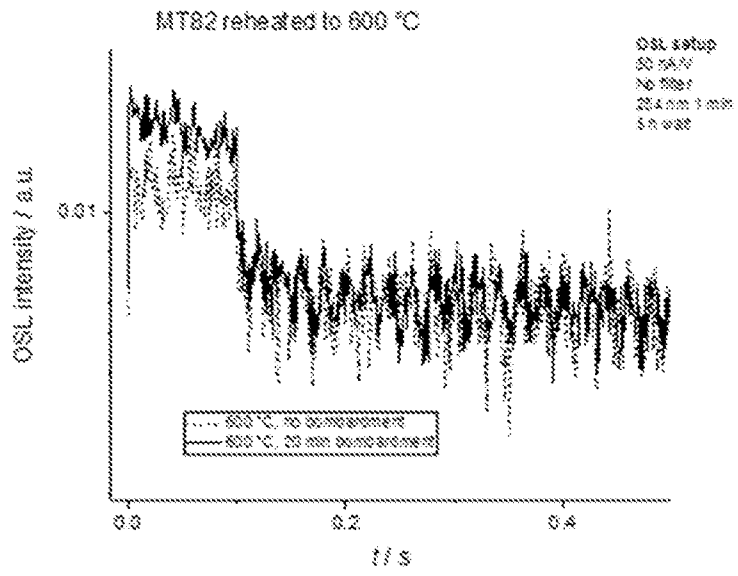


Fig. 11

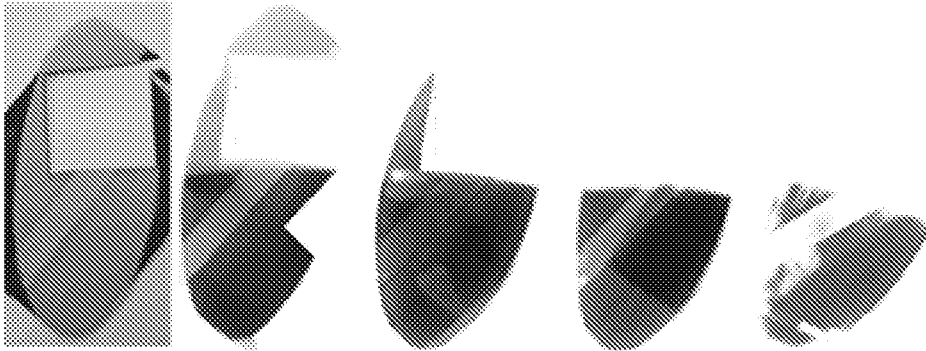


Fig. 12

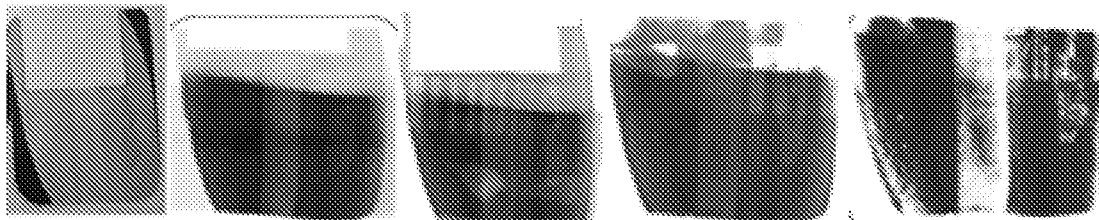


Fig. 13

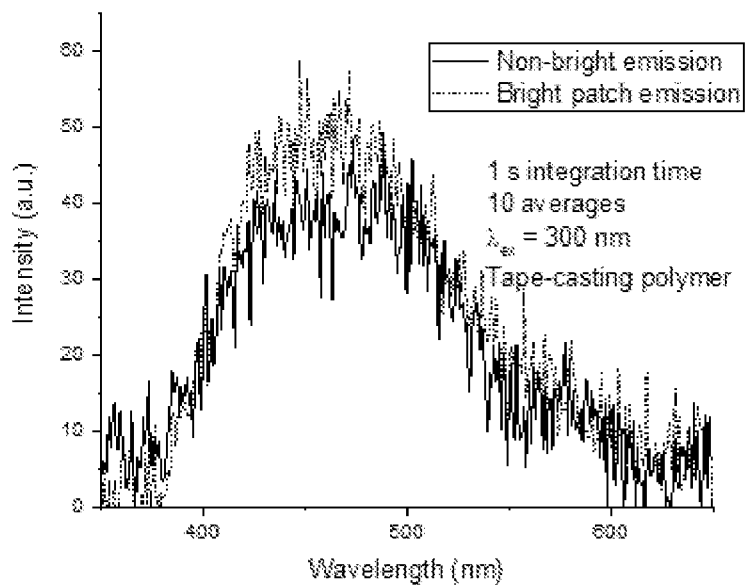


Fig. 14

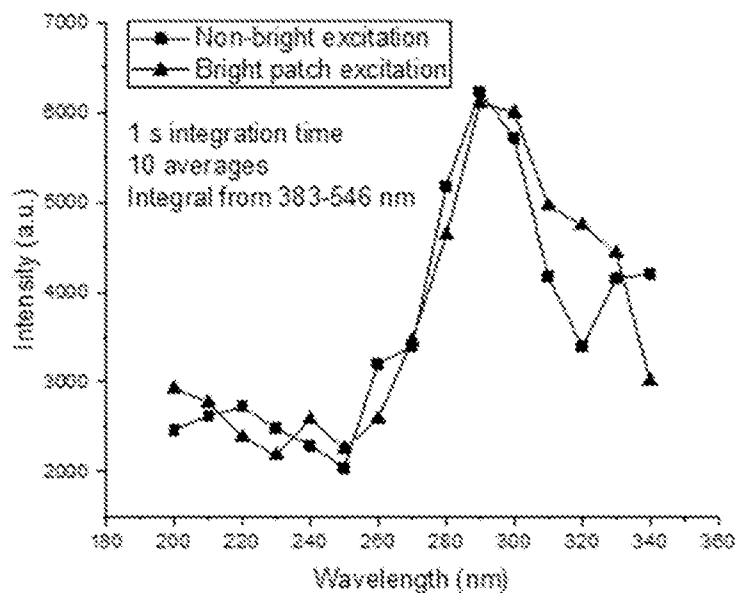


Fig. 15

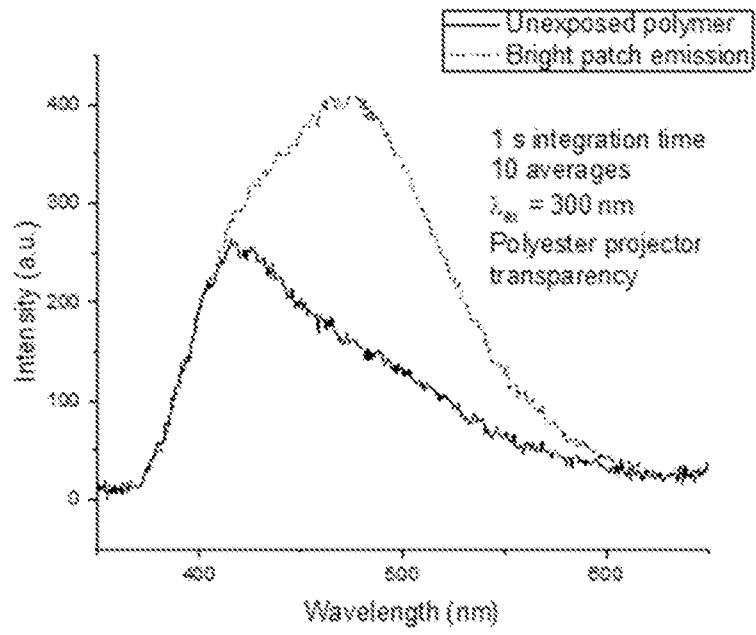


Fig. 16