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(54) Title: SCINTILLATOR UNIT FOR USE IN A TEMPERATURE DETECTION SYSTEM, METHOD AND SYSTEM FOR TEMPERATURE DETECTION, AND METHOD OF PRODUCING A SCINTILLATOR UNIT

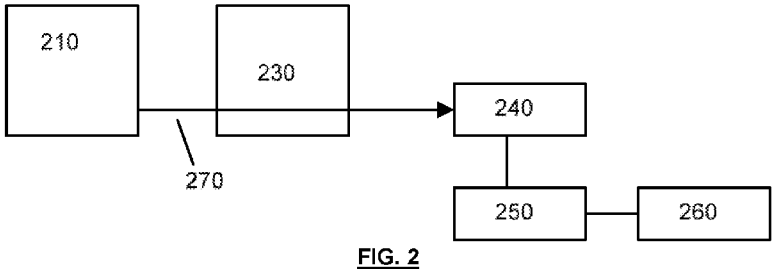

FIG. 2

(57) Abstract: A method comprises detecting scintillation from a scintillator unit including a scintillator; and determining a temperature of the scintillator or a sample in thermal contact with the scintillator, based on the detected scintillation, wherein a majority of the detected scintillation is excited by a radiation source provided integrally with the scintillator. A scintillator unit, system and method of producing a scintillator system are also provided.

Scintillator unit for use in a temperature detection system, method and system for temperature detection, and method of producing a scintillator unit.

[0001] This invention relates to a system and method for temperature detection, a scintillator unit for use in a temperature detection system, and a method of producing a scintillator unit.

BACKGROUND

[0002] According to some technologies, measurement of a sample temperature requires electrical leads between a sensor in thermal contact with an object and readout electronics. In some applications, such as those that involve motion of the object electrical connections may be inconvenient, impractical or even impossible. Such contact methods may also be unsuitable for applications such as furnaces, chemically aggressive environments, turbines, MRI, RF and microwave processes, or high-voltage equipment measurements.

[0003] Non-contact methods also exist, such as those based on exploiting a change of optical properties with temperature, such as refractive index, birefringence, transmission, light intensity, wavelength shift, or luminescence decay time. Herein, non-contact indicates that there is no a physical connection between readout electronics and a sensor.

[0004] Decay time thermometry (S. A. Allison and G. T. Gillies, Remote thermometry with thermographic phosphors: Instrumentation and application. *Rev. Sci. Instrum.* **68** (1997) 2615) exploits the temperature-dependence of the luminescence decay time constant of phosphorescent sensors. An example of a system for such measurements is illustrated schematically in Figure 1. A phosphorescent sensor 110, exhibiting a large change of the decay time constant in the temperature range of interest, is excited by a laser 120 via optical system 130. The luminance signal resulting from the excitation is delivered by the optical system 130 to a detector 140. The detector outputs the result of the detection to a DAQ 150 (data acquisition system), which provides the data to a computer 160 for data analysis to determine the temperature of the scintillator.

[0005] The laser may be a pulsed laser source, and the optical system may include flexible optical fibres or light guides. The detector may be a photo detector such as a Si-based diode or photomultiplier.

[0006] Decay time measurement does not rely on absolute intensities. Rather, temperature may be inferred from the distribution of the relative arrival times of individual photons. Thus, it is not necessary to require accurate reference measurements.

[0007] The present invention may address shortcomings, or provide improvements or alternatives to the current state of the art.

BRIEF SUMMARY OF THE DISCLOSURE

[0008] According to some aspects of the invention, a method comprises detecting scintillation from a scintillator unit including a scintillator; and determining a temperature of the scintillator or a sample in thermal contact with the scintillator, based on the detected scintillation, wherein a majority of the detected scintillation is excited by a radiation source provided integrally with the scintillator.

[0009] In some embodiments the radiation source may be internal to the scintillator. The radiation source may be chemically integrated into the scintillator. The radiation source may be an impurity in a matrix of the scintillator. The radioactive impurities forming the radiation source may be created in the scintillator by neutron irradiation of the scintillator. For example, radioactive impurities created in the scintillator by neutron irradiation of the scintillator may include ^{65}Zn, ^{110}Ag, ^{210}Bi, ^{210}Pb, ^{210}Po. Thus, the the radiation source may include one or more of these impurities. The scintillator and radiation source may be hermetically sealed. The scintillator and radiation source may be provided in a scintillator unit, such that substantially no radiation emitted by the radiation source is emitted from the scintillator unit.

[0010] According to some aspects a scintillator unit for use in a temperature detection system comprises a scintillator; and radiation source for causing scintillation, wherein the scintillator and radiation source are integral to the scintillator unit.

[0011] In some embodiments, the radiation source may be internal to the scintillator. The radiation source may be chemically integrated into the scintillator. The radiation source may be an impurity in a matrix of the scintillator. The impurity forming the radiation source may be created in the scintillator by neutron irradiation of the scintillator. The scintillator and radiation source may be hermetically sealed. The scintillator unit may be such that substantially no radiation emitted by the radiation source is emitted from the scintillator unit.

[0012] Some aspects provide a temperature detection system comprising a scintillator unit of an aspect or embodiment; and a scintillation detection section to detect scintillation, wherein the radiation source is configured to excite a majority of the scintillation emitted by the scintillator unit.

[0013] The temperature detection system may further comprise a processing section for receiving a detection result from the scintillation detection section and determining a temperature of the scintillator or a sample in thermal contact with the scintillator, based on the detected scintillation.

[0014] According to some aspects, a method of producing a scintillator unit having an integral radiation source comprises irradiating a scintillator with neutrons to form the radiation source as

radioactive impurities in the scintillator.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] Embodiments of the invention are further described hereinafter with reference to the accompanying drawings, in which:

Figure 1 illustrates a background temperature detection system.

Figure 2 illustrates a temperature detection system according to an example.

Figure 3 illustrates examples of variation of decay time constants with temperature.

Figure 4 illustrates some examples of scintillation units.

Figure 5 illustrates an example of a temperature detection system.

DETAILED DESCRIPTION

[0016] Figure 2 shows an example of a system according to an embodiment. A light signal (e.g. photons) from a scintillator of scintillator unit 210 is passed, via optical system 230, to a detector 240. The detector 240 outputs a signal indicative of detected photons to a data acquisition system 250 (DAQ), which provides data based on the signal to a computer 260 for data analysis to determine the temperature of the scintillator.

[0017] The scintillator unit 210 includes a scintillator and an excitation source in the form of a radiation source that is integrally provided in the scintillator unit 210. According to the present example, the radiation source takes the form of radioactive atoms or ions chemically integrated into the scintillator. In the present example, the radiation source is a plurality of radioactive ions or atoms present in the matrix of the scintillator as an impurity. In some examples the radiation source provides the scintillator unit with weak internal radioactivity (e.g. less than 10 kBq).

[0018] The scintillator may be a material doped with rare earth elements, Cr3+ or Mn3+, such as, CaF_2-Yb, SrF2-Yb , Y3Al5O12-Tb, Al2O3-Cr, Y3Al5O12-Cr, Y3Al5O12-Mn. The scintillator may be one of $CaWO_4$, CdWO4, Bi4Ge3O12, for example. The radiation source may be ^{241}Am, or radioactive impurities, such as ^{210}Pb, ^{210}Po, in the scintillator.

[0019] Where the scintillator unit 210 includes an integral radiation source, an external radiation source, such as a laser, is unnecessary. Accordingly, the cost and space required for the system can be reduced by omission of the laser and corresponding elements of the optical system. Moreover, there is no need to align the radiation source with the other elements of the system, simplifying setup and operation of the system.

[0020] In some examples, providing the radiation source in close proximity with the scintillator allows use of a relatively low level of radiation in the radiation source. Accordingly, in some

examples the radiation emitted by the radiation source or emitted from the scintillator unit 210 may permit the radiation source/scintillator unit 210 to be handled by a user without protective equipment, and without exceeding a safe dose. For example, the activity may be below the limit for classification as a radioactive source.

[0021] The scintillator may be of a material chosen for its properties, such as the temperature dependence of scintillation characteristics in the material. For example, where the temperature determination is to be based on decay time of the scintillation, the scintillator material may be based on the steepness of the decay time constant with temperature. A steep variation of decay time with temperature may be desirable. The temperature range of interest (i.e. the temperature range over which measurements are to be performed) may also influence the choice of scintillator material. For example, $CaWO_4$ is suitable for measurements over a specific temperature range (e.g. between 10 and 70 K). On the other hand, BGO is suitable for measurements over a broader temperature range (e.g. between 10K and 200K). Figures 3a and 3b show decay time variation with temperature for $CaWO_4$ and BGO, respectively. In some examples, the temperature detection system may be arranged to detect temperature at cryogenic temperatures. In some examples, the temperature detection system may be arranged to detect temperatures between 10K and 100 K.

[0022] The optical system 230 may include optical focusing and/or redirecting elements, such as an objective, an optical fiber, a mirror, a beam splitter, etc. In some examples an optical system 230 may be unnecessary.

[0023] Detector 240 may be a photomultiplier tube (PMT), with high gain and low jitter, for example. Detector types other than a PMT could alternatively be used, such as a Si-based diode or multipixel photon counter (MPPC). The detector 240 may be capable of counting individual photons in a scintillation event.

[0024] DAQ 250 may digitize the signal from the detector 240. The DAQ 250 may also perform preliminary analysis of the digitized detection signal (filtering, timestamping, etc). The DAQ 250 may include a field-programmable gate array (FPGA).

[0025] The DAQ 250 outputs data to computer 260. The data may include the digitized signal, and/or information derived therefrom. The computer 260 may be any device or system suitable for processing the data. Computer 260 may be a desktop PC, a laptop PC, or may be a notebook, netbook or tablet computing device. Computer 260 may include a processor, a storage device, input and output devices, etc. The storage device may store data and/or software. The software may include an operating system and/or data analysis software.

[0026] Figure 4 illustrates examples of scintillator units having integral scintillators and radiation sources. Figure 4a schematically illustrates a scintillator unit including a scintillator

310 having radioactive ions or atoms as impurities 320 within the matrix of the scintillator, the radioactive impurities forming the source of the radiation.

[0027] Figure 4b illustrates an example in which the scintillator unit includes a scintillator 330 surrounding a radiation source 340. In this example the radiation source 340 is internal to the scintillator 330.

[0028] Figure 4c illustrates an example in which the scintillator unit includes a scintillator 350 and a radiation source 360 located proximally to the scintillator350. In the example of Figure 4c the radiation source 360 is in physical contact with the scintillator 350.

[0029] In some examples, the radiation source may be chemically bonded to or chemically integrated into the scintillator.

[0030] In some cases radioactive impurity forming the radiation source may be created in a scintillator by neutron irradiation of the scintillator itself.

[0031] In some examples the scintillator unit may include a casing or housing 370. The housing may be arranged such that essentially no radiation from the radiation source is emitted from the scintillator unit. Herein essentially no radiation may mean a radiation level below a threshold for classification as a radiation source for the purposes of laboratory safety. In some examples a material of the housing and a thickness of the material are arranged such that essentially no radiation from the radiation source is emitted from the scintillator unit.

[0032] In some examples the scintillator unit is hermetically sealed.

[0033] In some examples the type of radiation source, amount/concentration of radiation source, and geometry of the scintillator and radiation source is such that essentially no radiation may be emitted from the scintillator unit. In some examples, the level of emitted radiation from the scintillator unit is such that the scintillator unit is not classed as a radiation source for safety purposes.

[0034] In some examples the radiation source is an alpha source (e.g. ^{210}Po). In some examples, the radiation source emits mixed alpha and gamma radiation only of a low energy (e.g. ^{210}Pb, ^{241}Am).

[0035] Figure 5 shows an example arrangement, suitable for use at a synchrotron based beamline having a camera 570 and optical system (530-535) for visual inspection of a sample (such as a protein crystal, for example). A sample holder 510, is in thermal contact with a scintillation unit 520 (such as one of the scintillation units in Figure 4). Photomultiplier tube 540 is provided to receive photons from the scintillator 520 via the optical system (530-535). The output of the photomultiplier tube 540 is provided to DAQ 550 and the output from the DAQ 550 is received and analyzed using PC 560, as described in relation to Figure 2.

[0036] The optical system of Figure 5 includes lenses 530, mirrors 532 and focusing mirror 535. The focusing mirror 535 is moveable, to direct light from the sample to the camera 570, or direct photons from the scintillator unit 520 to the photomultiplier tube 540.

[0037] The lenses 530 and mirrors 532 may form a pre-existing optical system of a beamline, e.g. for visualization of the sample. For example, Figure 5 shows a viewing system. Essentially the same optical elements (i.e. with the omission of the lens closest to the camera and the addition of the focusing mirror 535) may be used to deliver the photons from the scintillator unit 520 to the PMT. Using elements of a pre-existing optical system for delivering photons to the photomultiplier tube may simplify and reduce the cost of adding the temperature detection system to an existing experimental setup that already has a viewing system in place.

[0038] Using at least some optical elements for both the visualization system and the temperature detection system may reduce, or provide efficiencies in, the size and cost of the system.

[0039] In some examples the temperature detection system may be suitable for use in a beamline, such as a beamline of a synchrotron source, spallation source, research reactor, or particle accelerator. Some examples may be suitable for use in microfocusing beamlines, which may have congested experimental space and temperature can change dramatically within a short distance.

[0040] Herein, references to determining a temperature of a scintillator also include estimating or determining a temperature of a sample in thermal contact with the scintillator.

[0041] Using a decay time measurement technique, as described in the examples above, may reduce or obviate reliance on absolute intensities, which may reduce a need for accurate reference measurements. However, other techniques may also be applied, either as alternatives to or in combination with a decay time technique, based on other temperature dependent properties of the luminescence.

[0042] In some examples the radiation source is the only excitation source for the scintillator. However, in some cases, additional, external radiation may also be unavoidable. According to some examples the majority of the scintillation emitted by the scintillation unit is due to the radiation source. In some examples, the majority of the scintillation detected by the detector is due to the radiation source. Herein, the majority of the scintillation means to 50% or more of the scintillation. In some examples the scintillation due to the radiation source is at least 80% of the scintillation.

[0043] Some examples provide a radiation unit having an integral scintillator for use as a temperature sensor. The radiation unit has the same components and can be produced in the same manner as the scintillation unit described above.

[0044] Throughout the description and claims of this specification, the words “comprise” and “contain” and variations of them mean “including but not limited to”, and they are not intended to (and do not) exclude other moieties, additives, components, integers or steps. Throughout the description and claims of this specification, the singular encompasses the plural unless the context otherwise requires. In particular, where the indefinite article is used, the specification is to be understood as contemplating plurality as well as singularity, unless the context requires otherwise.

[0045] Features, integers, characteristics, compounds, chemical moieties or groups described in conjunction with a particular aspect, embodiment or example of the invention are to be understood to be applicable to any other aspect, embodiment or example described herein unless incompatible therewith. All of the features disclosed in this specification (including any accompanying claims, abstract and drawings), and/or all of the steps of any method or process so disclosed, may be combined in any combination, except combinations where at least some of such features and/or steps are mutually exclusive. The invention is not restricted to the details of any foregoing embodiments. The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any method or process so disclosed.

[0046] The reader's attention is directed to all papers and documents which are filed concurrently with or previous to this specification in connection with this application and which are open to public inspection with this specification, and the contents of all such papers and documents are incorporated herein by reference.

CLAIMS

1. A method comprising:

detecting scintillation from a scintillator unit including a scintillator; and

determining a temperature of the scintillator or a sample in thermal contact with the scintillator, based on the detected scintillation, wherein

a majority of the detected scintillation is excited by a radiation source provided integrally with the scintillator.

2. The method of claim 1, wherein the radiation source internal to the scintillator.

3. The method of claim 2, wherein the radiation source is chemically integrated into the scintillator.

4. The method of claim 3, wherein the radiation source is an impurity in a matrix of the scintillator.

5. The method of claim 4, wherein the impurity forming the radiation source is created in the scintillator by neutron irradiation of the scintillator.

6. The method of any preceding claim, wherein the scintillator and radiation source are hermetically sealed.

7. The method of any preceding claim, wherein the scintillator and radiation source are provided in a scintillator unit, and substantially no radiation emitted by the radiation source is emitted from the scintillator unit.

8. A scintillator unit for use in a temperature detection system, the scintillator unit comprising:

a scintillator; and

radiation source for causing scintillation of the scintillator, wherein

the scintillator and radiation source are integral to the scintillator unit.

9. The scintillator unit of claim 8, wherein the radiation source internal to the scintillator.

10. The scintillator unit of claim 9, wherein the radiation source is chemically integrated into the scintillator.

11. The scintillator unit of claim 10, wherein the radiation source is an impurity in a matrix of the scintillator.

12 The scintillator unit of claim 11, wherein the impurity forming the radiation source is created in the scintillator by neutron irradiation of the scintillator.

13. The scintillator unit of any one of claims 8 to 12, wherein the scintillator and radiation source are hermetically sealed.

14. The scintillator unit of any one of claims 8 to 13, wherein substantially no radiation emitted by the radiation source is emitted from the scintillator unit.

15. A temperature detection system comprising:
the scintillator unit of any one of claims 8 to 14; and
a scintillation detection section to detect scintillation, wherein
the radiation source is configured to excite a majority of the scintillation emitted by the scintillator unit.

16. The temperature detection system according to claim 15, further comprising:
a processing section for receiving a detection result from the scintillation detection section and determining a temperature of the scintillator or a sample in thermal contact with the scintillator, based on the detected scintillation.

17. A method of producing a scintillator unit having an integral radiation source, the method comprising: irradiating a scintillator with neutrons to form the radiation source as radioactive impurities in the scintillator.

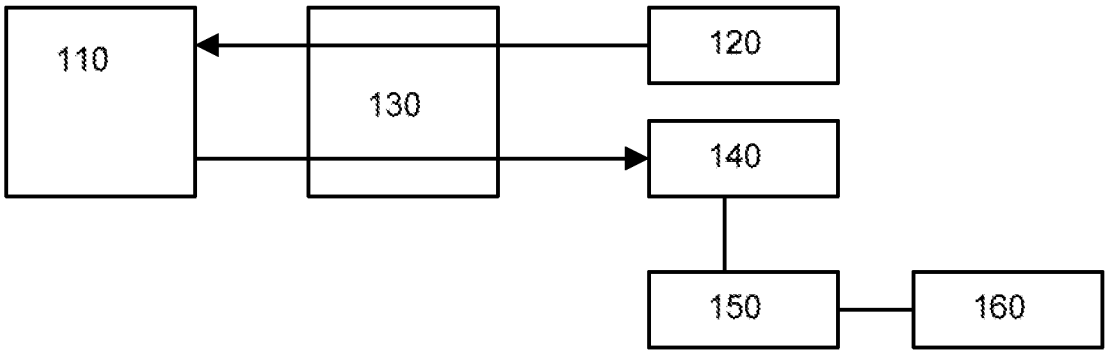

FIG. 1

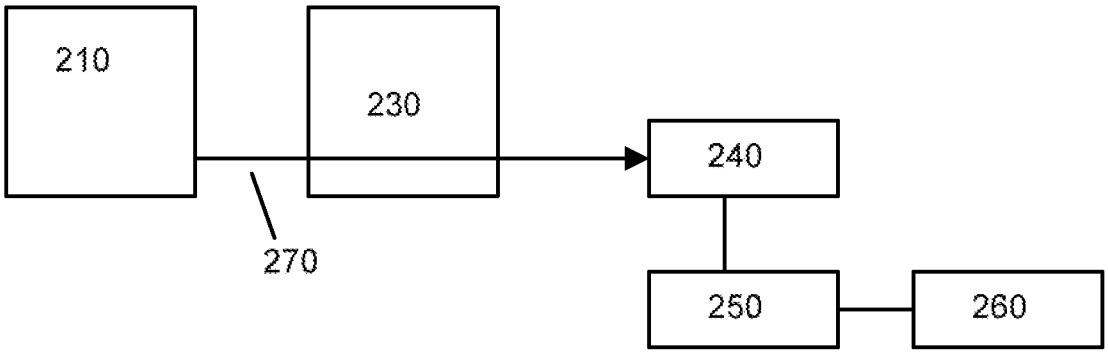

FIG. 2

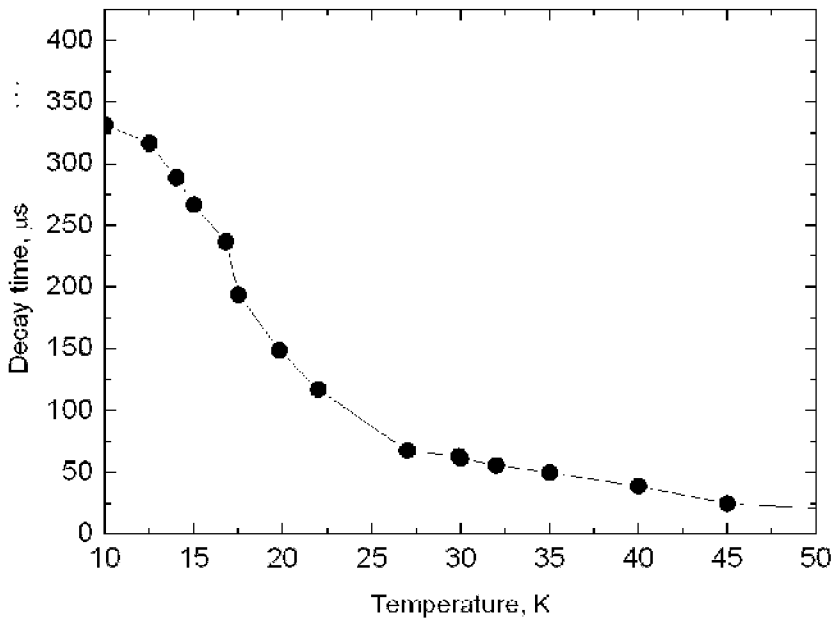

FIG. 3a

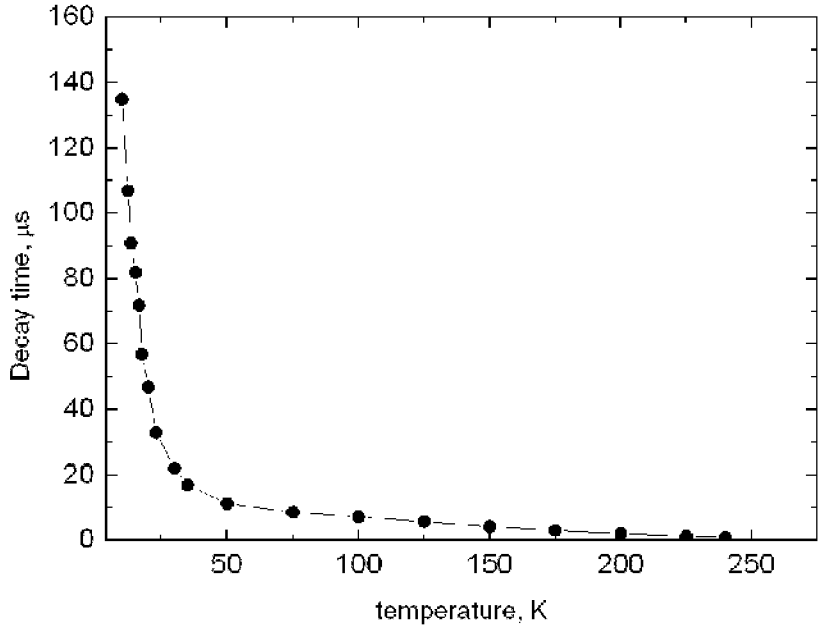

FIG. 3b

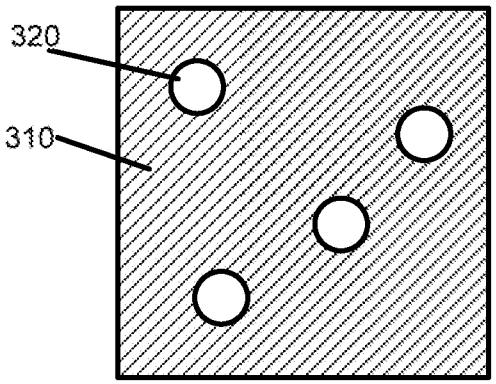

FIG. 4a

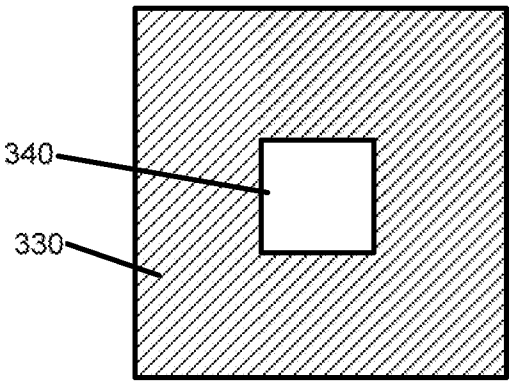

FIG. 4b

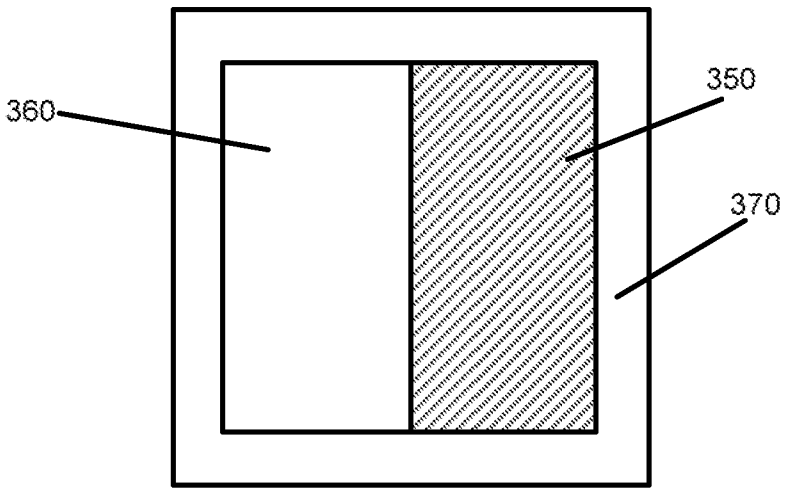

FIG. 4c

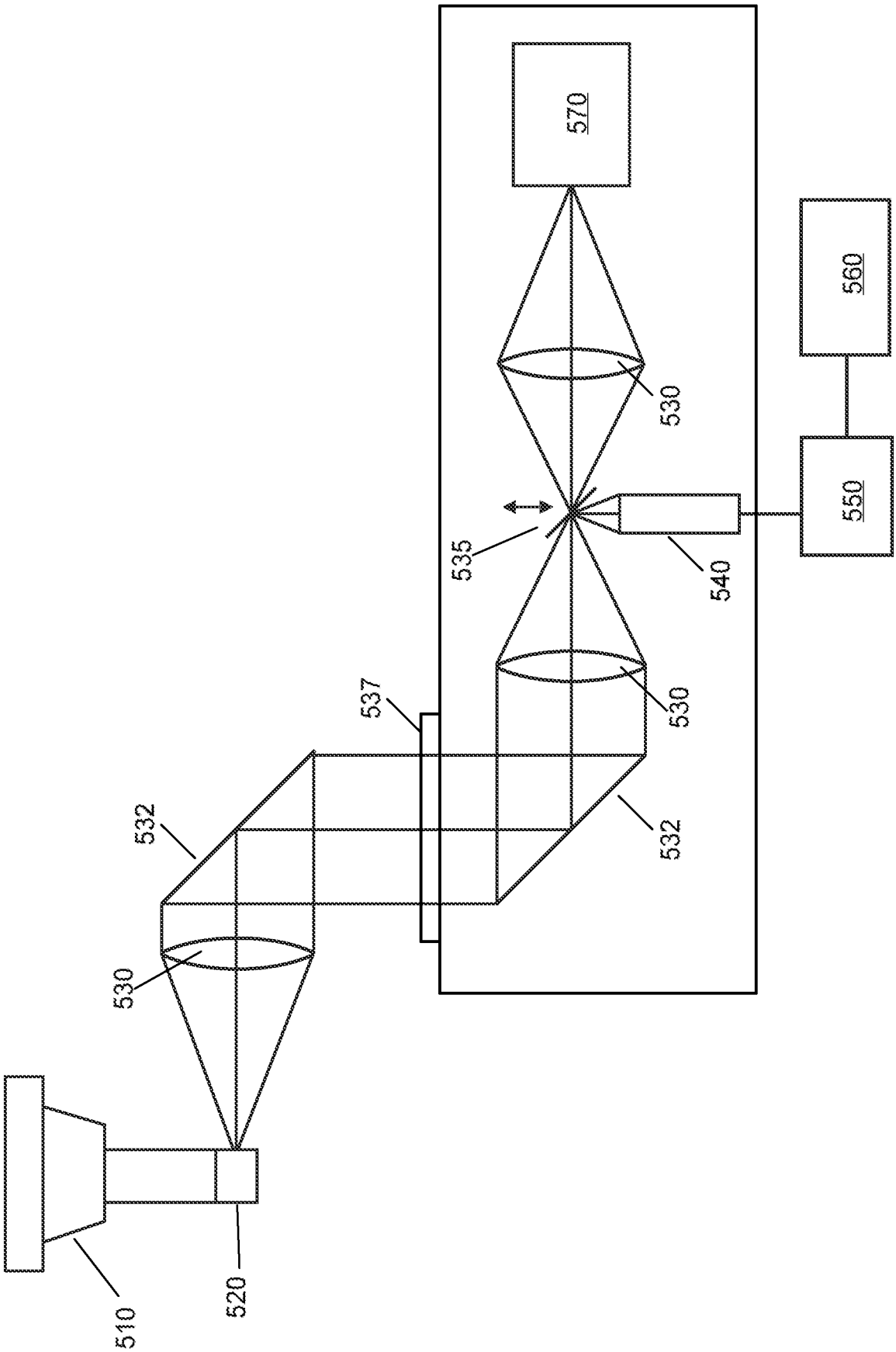

FIG. 5

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2014/050766

A. CLASSIFICATION OF SUBJECT MATTER
INV. G01K11/20
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)
G01K

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 practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 245 507 A (SAMULSKI T V) 20 January 1981 (1981-01-20) column 6, line 52 - column 7, line 19; claims 20-24; figure 3 ----- -/--	1-17

[X] Further documents are listed in the continuation of Box C.

[X] 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 application or patent 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)

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"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	Date of mailing of the international search report
25 July 2014	01/08/2014

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Rosello Garcia, M

2

Form PCT/ISA/210 (second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2014/050766

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	MAEKAWA F ET AL: "Development of whole energy absorption spectrometer for decay heat measurement", NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH. SECTION A: ACCELERATORS, SPECTROMETERS, DETECTORS, AND ASSOCIATED EQUIPMENT, ELSEVIER BV * NORTH-HOLLAND, NL, vol. 450, no. 2-3, 11 August 2000 (2000-08-11), pages 467-478, XP004215584, ISSN: 0168-9002, DOI: 10.1016/S0168-9002(00)00301-6 the whole document -----	1-17
A	S W Allison ET AL: "Remote thermometry with thermographic phosphors: Instrumentation and applications", Rev. Sci. Instrum. 68 (7), July 1997, 1 July 2007 (2007-07-01), pages 2615-2650, XP055131261, Retrieved from the Internet: URL:https://engineering.purdue.edu/people/john.p.sullivan.1/rdtphosphors2.pdf [retrieved on 2014-07-23] cited in the application the whole document -----	1-17
A	US 5 885 484 A (ALLISON STEPHEN W [US] ET AL) 23 March 1999 (1999-03-23) the whole document -----	1-17

2

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/GB2014/050766

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4245507 A	20-01-1981	NONE	
US 5885484 A	23-03-1999	US 5730528 A	24-03-1998
		US 5885484 A	23-03-1999