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(54) **Title:** ILLUMINATING DEVICE COMBINING A LED AND A DIFFUSING SHEET

(57) **Abstract:** The invention relates to a luminous device comprising at least one LED having a luminous flux of greater than 3 Lm, advantageously greater than 5 Lm, preferably greater than 10 Lm and even more preferably greater than 50 Lm, and to a cover made of a transparent plastic in which scattering particles with a content of 3 to 30% are dispersed. The scattering particles may be of organic nature. In which case they may be polyamide or PTFE particles, methyl-methacrylate-based crosslinked particles, crosslinked styrene-based particles or silicone particles. The scattering particles may be of mineral nature. In this case they may be $BaSO_4$, TiO_2, ZnO, $CaCO_3$, MgO or Al_2O_3 particles or hollow glass microspheres. The LED may be a colour LED or a white LED.

ILLUMINATING DEVICE COMBINING A LED AND A DIFFUSING SHEET

The present invention relates to a luminous device comprising at least one light-emitting diode and at least one cover made of a transparent plastic in which particles that scatter the light emitted by the light-emitting diode are dispersed.

Light-emitting diodes or LEDs are increasingly used in everyday life. They allow conventional artificial light sources, such as incandescent lamps and neon tubes, to be effectively replaced as they consume less electrical power and can operate at low voltages (12 V for example). Furthermore, they are more compact and have very long lifetimes, which may range up to 100 000 hours of operation. Although the discovery of red LEDs goes back to the 1960s, many years had to elapse before the appearance of LEDs having sufficient illumination and before the development of LEDs of different colours. At the present time, there are LEDs that emit in the red, the green or the blue, white LEDs, and LEDs that emit in the ultraviolet or infrared spectrum. Extensive research has also allowed the light flux emitted by LEDs to be significantly increased. LEDs having a light flux in excess of 3-5 lumens (Lm) have already been marketed and certain recent developments suggest that this may reach, or even exceed, 100 lumens.

Light devices that integrate one more LEDs are, for example, motor vehicle illumination devices (at the front or rear), indicating panels, luminous displays, spotlights, street lighting, box-letters, etc.

All these luminous devices consist of a light source and a cover made of a plastic whose function is to mask and protect the light source, while still ensuring good transmission of the light emitted by the light source. The plastic may be coloured or may have decorative elements or patterns. The cover also has the function of scattering the emitted light so that the illumination is softened and not dazzling. The scattering of the light emitted by the light source is achieved by dispersing scattering particles of organic or mineral nature in the plastic.

Replacing a conventional light source with an LED results in a modification of the illumination. This is because an LED, especially an LED having a high luminous flux, exhibits directional illumination whereas for example the illumination of a neon tube is from 0 to 360°. In addition, the emission spectrum of an LED is completely different from that of a conventional light source. The development of luminous devices incorporating one or more LEDs with a luminous flux of greater than 3 Lm therefore requires the plastic cover to be adapted.

The Applicant has found that a luminous device comprising at least one LED having a luminous flux of greater than 3 Lm and a cover made of a transparent plastic in which from 3 to 30% of scattering particles are dispersed allows the problem posed to be solved.

[Prior art]

US 6 723 772 discloses a scattering composition comprising a transparent plastic, based on methyl methacrylate or styrene, and organic or mineral scattering particles. The particle content is between 0.1 and 20% by weight, or expressed differently between 1 and 500 g/m^2.

US 2004/0191550 discloses a sheet comprising a layer (A) composed of a resin based on methyl methacrylate and one or two layers (B) composed of a blend of a resin based on methyl methacrylate and a resin based on vinylidene fluoride. The sheet may contain organic or mineral scattering particles in the layer (A) and be illuminated by a neon lamp or by an LED.

EP 1 369 224 discloses a coextruded sheet comprising at least two layers - a layer of a thermoplastic filled with organic scattering particles and a layer of a transparent thermoplastic. The sheet may be illuminated by an LED. Within the context of the present invention, the process for manufacturing the cover that is illuminated by the LED is simpler as the cover is not manufactured by coextrusion but by simple extrusion or else by the casting process in the case of PMMA. The cover according to the invention therefore has only a single layer.

WO 03/052315 discloses a luminous device that combines a cover made of a transparent plastic and an LED. The transparent plastic contains 1.5 to 2.5% by weight of $BaSO_4$ or polystyrene particles. In the case of crosslinked plastic particles, the content varies from 0.1 to 10% by weight.

These documents make no reference to the combination of an LED having a high luminous flux, greater than 3 Lm, and a transparent plastic comprising by weight 3 to 30% of scattering particles.

[Brief description of the invention]

The invention relates to a luminous device comprising at least one LED having a luminous flux of greater than 3 Lm, advantageously greater than 5 Lm, preferably greater than 10 Lm and even more preferably greater than 50 Lm, and a cover made of a transparent plastic in which scattering particles with a content of 3 to 30%, preferably 5 to 30%, most preferably 5 to 10% are dispersed.

The invention also relates to the uses of this luminous device.

The invention will be more clearly understood on reading the following detailed description, from the non-limiting examples of embodiments thereof and on examining the appended figures.

[Figures]

Figure 1 shows the diagram of an LED **1**. This consists of an anode **2**, a cathode **3** and a conducting wire **4** which allow the semiconductor **5** to be powered, the semiconductor itself being placed on a light-reflecting dish **6**. A protective shell **7** made of a transparent plastic protects the semiconductor and transmits the emitted light.

Figure 2 shows the emission spectrum (relative intensity in % as a function of the wavelength in nm) characteristic of a colour LED.

Figure 3 shows the emission spectrum of a white LED (relative intensity in % as a function of the wavelength in nm) that uses the re-emission of a phosphorescent compound.

Figure 4 shows the typical radiation profile of an LED (relative intensity in % as a function of the angular displacement θ in °).

Figure 5 shows an example of a luminous device. This is a letter **8** (an A) of a light sign illuminated by LEDs **1**. To simplify matters, the electrical power supply wires have not been shown in the figure. The letter A includes, on the front face, a cover **9** that transmits the light emitted by the LEDs, while masking and protecting the LEDs. The other faces of the letter **8**, for example the face **10**, are opaque.

Figure 6 shows the x/y chromaticity diagram, the achromatic point A being indicated therein by coordinates $x_A = 1/3$ and $y_A = 1/3$. A colour LED is shown in this diagram by the point B having coordinates x_B and y_B. The line D connects the point A with the point B. The light from the colour LED transmitted by a coloured cover is characterized by the point T. The point T must lie within a rectangle having the point B as orthocentre. This rectangle has a half-length L that corresponds to 0.2 x/y units and a half-width l that corresponds to 0.05 x/y units.

[Detailed description]

The term "transparent plastic" denotes a plastic of thermoplastic or thermosetting type, having a light transmission in the visible range of at least 50%, preferably at least 70% and even more preferably at least 80% according to the DIN 67-507 standard (this is the light transmission of the transparent plastic with no scattering particle; when the scattering particles are dispersed in the transparent plastic, the transparency decreases). By way of example, the transparent plastic may be crystal polystyrene, polyethylene terephthalate (PET), a transparent, especially clarified, polyolefin, for example clarified polypropylene, PMMA, a transparent polyamide or polycarbonate.

PMMA and polycarbonate are the two transparent plastics of choice because they are easy to process, are commercially available and have a high transparency. In addition, these two plastics exhibit excellent thermomechanical strength, allowing compact luminous devices to be produced. This is because, although the energy efficiency of an LED (that is to say the efficiency of converting electrical energy into light energy) is much better than for an incandescent lamp, some of the energy is nevertheless converted into heat. In the case of compact luminous devices, this heat builds up and rapidly raises the temperature inside the device.

The term "PMMA" denotes a methyl methacrylate (MMA) homopolymer or a copolymer comprising from 80 to 99.5% by weight of methyl methacrylate and from 0.5 to 20% by weight of at least one monomer having at least one ethylenic unsaturation that can copolymerize with methyl methacrylate. These monomers are well known and mention may be made, in particular, of styrene, alpha-methylstyrene, acrylic and methacrylic acids and alkyl-(meth)acrylates in which the alkyl group has from 2 to 4 carbon atoms. As examples, mention may be made of methyl acrylate and ethyl, butyl or 2-ethylhexyl (meth)acrylate.

Advantageously, this is a methyl methacrylate homopolymer or a copolymer comprising, by weight, 90 to 99.7%, preferably 90 to 99.5%, of methyl methacrylate and 0.3 to 10%, preferably 0.5 to 10%, of at least one monomer having at least one ethylenic unsaturation that can be copolymerized with methyl methacrylate. Preferably, the comonomer is methyl acrylate or ethyl acrylate.

PMMA is a transparent plastic appreciated in particular because it possesses excellent ageing resistance, especially resistance to ultraviolet radiation, and good scratch resistance.

The PMMA may contain an impact modifier, which improves the impact resistance thereof. The impact modifier is in the form of fine particles having an elastomer core and at least one thermoplastic shell, the size of the particles being in general less than 1 µm and advantageously between 50 and 300 nm. The impact modifier is prepared by emulsion polymerization. The impact modifier content in the PMMA is between 0 and 20%, preferably between 0 and 10%, by weight.

The core may for example consist of:

- an isoprene or butadiene homopolymer; or
- isoprene copolymers with at most 30 mol% of a vinyl monomer; or
- butadiene copolymers with at most 30 mol% of a vinyl monomer.

The vinyl monomer may be styrene, an alkyl styrene, acrylonitrile or an alkyl(meth)acrylate.

The core may also consist of:

- an alkyl(meth)acrylate homopolymer; or
- copolymers of an alkyl(meth)acrylate with at most 30 mol% of a monomer chosen from another alkyl(meth)acrylate and a vinyl monomer.

The alkyl(meth)acrylate is advantageously butyl acrylate. The vinyl monomer may be styrene, an alkyl styrene, acrylonitrile, butadiene or isoprene.

Advantageously, the core may be completely or partly crosslinked. It is sufficient to add at least difunctional monomers during the preparation of the core. These monomers may be chosen from poly(meth)-acrylic esters of polyols, such as butylene di(meth)-acrylate and trimethylolpropane trimethacrylate. Other difunctional monomers may for example be divinyl-benzene, trivinylbenzene, vinyl acrylate and vinyl methacrylate. The core may also be crosslinked by introducing into it, by grafting or as comonomer during the polymerization, unsaturated functional monomers such as unsaturated carboxylic acid anhydrides, unsaturated carboxylic acids and unsaturated epoxides. As examples, mention may be made of maleic anhydride, (meth)acrylic acid and glycidyl methacrylate.

The shell or shells are styrene, alkyl styrene or methyl methacrylate homopolymers or copolymers containing at least 70 mol% of one of these monomers mentioned above and at least one comonomer chosen from the other monomers mentioned above, another alkyl-(meth)acrylate, vinyl acetate and acrylonitrile. The shell may be functionalized by introducing thereinto, by grafting or as comonomer during polymerization, unsaturated functional monomers such as unsaturated carboxylic acid anhydrides, unsaturated carboxylic acids and unsaturated epoxides. As examples, mention may be made of maleic anhydride, (meth)acrylic acid and glycidyl methacrylate.

As examples of impact modifiers, mention may be made of core-shell copolymers having a polystyrene shell and core-shell copolymers having a PMMA shell. There are also core-shell copolymers having two shells, one made of polystyrene and the other on the outside made of PMMA. Examples of impact modifiers and their method of preparation are described in the following patents: **US 4 180 494, US 3 808 180, US 4 096 202, US 4 260 693, US 3 287 443, US 3 657 391, US 4 299 928, US 3 985 704** and **US 5 773 520**.

Advantageously, the core represents 70 to 90% and the shell 30 to 10% by weight of the impact modifier.

The impact modifier may be of the soft/hard type. As an example of an impact modifier of the soft/hard type, mention may be made of that consisting:

(i) of 75 to 80 parts of a core comprising at least 93 mol% of butadiene, 5 mol% of styrene and 0.5 to 1 mol% of divinylbenzene and

(ii) of 25 to 20 parts of two shells essentially of the same weight, the inner one made of polystyrene and the other outer one made of PMMA.

As another example of a soft/hard type impact modifier, mention may be made of that having a poly(butyl acrylate) or butyl acrylate/butadiene copolymer core and a PMMA shell.

The impact modifier may also be of the hard/soft/hard type, that is to say it contains, in this order, a hard core, a soft shell and a hard shell. The hard parts may consist of the polymers of the shell of the above soft/hard copolymers and the soft part may consist of the polymers of the core of the above soft/hard copolymers.

Mention may be made, for example, of an impact modifier of the hard/soft/hard type consisting:

(i) of a core made of a methyl methacrylate/ethyl acrylate copolymer;

(ii) of a layer made of a butyl acrylate/styrene copolymer;

(iii) of a shell made of a methyl methacrylate/ethyl acrylate copolymer.

The impact modifier may also be of the hard (core)/soft/semi-hard type. In this case the "semi-hard" outer shell consists of two shells, one being the intermediate shell and the other the outer shell. The intermediate shell is a copolymer of methyl methacrylate, styrene and at least one monomer chosen from alkyl acrylates, butadiene and isoprene. The outer shell is a PMMA homopolymer or copolymer.

An example of a hard/soft/semi-hard impact modifier is that consisting, in this order:

(i) of a core made of a methyl methacrylate/ethyl acrylate copolymer;

(ii) of a shell made of a butyl acrylate/styrene copolymer;

(iii) of a shell made of a methyl methacrylate/butyl acrylate/styrene copolymer; and

(iv) of a shell made of a methyl methacrylate/ethyl acrylate copolymer.

The PMMA may also contain at least one anti-UV additive that increases the lifetime of the PMMA. The PMMA may contain other additives that are well known to those skilled in the art. These may for example be an antistatic agent, a flame retarder, an antioxidant, etc.

The term **"polycarbonate (PC)"** denotes a polyester of carbonic acid, that is to say a polymer obtained by the reaction of at least one carboxylic acid derivative with at least one aromatic or aliphatic diol. The preferred aromatic diol is bisphenol A, which reacts with phosgene or else, by transesterification, with ethyl carbonate.

It may be a homopolycarbonate or copolycarbonate based on a diphenol of formula HO-Z-OH for which Z denotes a divalent organic radical possessing from 6 to 30 carbon atoms and containing one or more aromatic group(s). As examples, the diphenol can be:

- dihydroxybiphenyls,
- bis(hydroxyphenyl)alkanes,
- bis(hydroxyphenyl)cycloalkanes,
- indane bisphenols,
- bis(hydroxyphenyl) ethers,
- bis(hydroxyphenyl) ketones,
- bis(hydroxyphenyl) sulphones,
- bis(hydroxyphenyl) sulphoxides,
- α,α'-bis(hydroxyphenyl)diisopropylbenzenes.

It may also relate to derivatives of these compounds obtained by alkylation or halogenation of the aromatic ring. Mention will more particularly be made, among the compounds of formula HO-Z-OH, of the following compounds:

- hydroquinone,
- resorcinol,
- 4,4'-dihydroxybiphenyl,
- bis(4-hydroxyphenyl) sulphone,
- bis(3,5-dimethyl-4-hydroxyphenyl)methane,
- bis(3,5-dimethyl-4-hydroxyphenyl) sulphone,

- 1,1-bis(3,5-dimethyl-4-hydroxyphenyl)-para/meta-isopropylbenzene,
- 1,1-bis(4-hydroxyphenyl)-1-phenylethane,
- 1,1-bis(3,5-dimethyl-4-hydroxyphenyl)cyclohexane,
- 1,1-bis(4-hydroxyphenyl)-3-methylcyclohexane,
- 1,1-bis(4-hydroxyphenyl)-3,3-dimethylcyclohexane,
- 1,1-bis(4-hydroxyphenyl)-4-methylcyclohexane,
- 1,1-bis(4-hydroxyphenyl)cyclohexane,
- 1,1-bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane,
- 2,2-bis(3,5-dichloro-4-hydroxyphenyl)propane,
- 2,2-bis(3-methyl-4-hydroxyphenyl)propane,
- 2,2-bis(3,5-dimethyl-4-hydroxyphenyl)propane,
- 2,2-bis(4-hydroxyphenyl)propane (or bisphenol A),
- 2,2-bis(3-chloro-4-hydroxyphenyl)propane,
- 2,2-bis(3,5-dibromo-4-hydroxyphenyl)propane,
- 2,4-bis(4-hydroxyphenyl)-2-methylbutane,
- 2,4-bis(3,5-dimethyl-4-hydroxyphenyl)-2-methylbutane,
- α,α'-bis(4-hydroxyphenyl)-o-diisopropylbenzene,
- α,α'-bis(4-hydroxyphenyl)-m-diisopropylbenzene (or bisphenol M).

The preferred polycarbonates are the homopolycarbonates based on bisphenol A or 1,1-bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane and the copolycarbonates based on bisphenol A and 1,1-bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane.

The polycarbonates are produced by reaction of diphenol(s) with a carbonate by transesterification. For further details, reference may be made to Schnell, "Chemistry and Physics of Polycarbonates", Interscience Publishers, 1964, or alternatively to "Polycarbonates" in Encyclopedia of Polymer Science and Engineering, Volume 11, 2nd edition, 1988, pages 648-718, and in

U. Grigo, K. Kircher and P.R. Müller, "Polycarbonate", in the work Becker, Braun, Kunststoff-Handbuch, Volume 3/1, Polycarbonate, Polyacetale, Polyester, Celluloseester, Carl Hanser Verlag Munich, Vienna 1992, pages 117 to 299.

Polycarbonate is a transparent plastic appreciated especially because it possesses excellent impact strength and for its fire-retardant properties.

Type of LED

An LED is a light source that exploits the physical principle of electroluminescence, that is to say the emission of light by a semiconductor when a potential difference is applied to a junction of the p-n type. For further details, the reader may refer to Ullmann's Encyclopaedia of Industrial Chemistry, Volume A9, 5th edition, "Electroluminescent Materials and Devices", pages 255-264 or to Chapter 7 of the work by S.M. Sze "Modern Semiconductor Device Physics" published by Wiley (ISBN: 0471152374) in 1997. The LED therefore comprises the semiconductor itself and also other elements for the operation and handling of the LED, such as a transparent protective shell, supply contacts (anode/cathode), optionally a dish that reflects the emitted light, etc. According to the invention, the scattering particles are not dispersed in the transparent protective shell but in the cover.

Among LEDs that emit in the visible spectrum, distinction may be made between colour LEDs and white LEDs.

The emission spectrum of a colour LED is very narrow and has a mid-height width of between 20 and 55 nm, preferably between 30 and 50 nm (as may be seen in Figure 2). The emission spectrum depends on the nature of the p-n junction, and therefore on the chemical nature of material used for the semiconductor. The following equation connects the wavelength λ (in nm) of the emitted light to the bandgap E_{band} (in eV), which depends on the semiconductor material used:

$$\lambda = \frac{hc}{E_{band}}$$

where c denotes the velocity of light (3 × 10^8 m/s) and h denotes Planck's constant (4.136 × 10^{-15} eV/s).

For example, if the semiconductor material is gallium arsenide GaAs, the bandgap is 1.35 eV and the LED emits in the near infrared at around 920 nm. Gallium phosphide arsenide GaAsP, gallium phosphide GaP, gallium nitride GaN and gallium indium nitride GaInN are other examples among other intermetallic compounds that can be used for manufacturing an LED.

The emission of white light by an LED is more problematic as it is necessary to combine the three essential components of white light, namely blue, red and green. It is also necessary to mix these three components so as to obtain a white light having good colour rendition or CRI and a good colour temperature (in kelvin). At the present time, there are at least three technologies for obtaining emission of white light:

- the first consists in combining an LED emitting in the blue with one or more phosphorescent compounds that reemit in the yellow. This is a method that is widely used at the present time, but the CRI (colour rendition index) is poor, less than 75, and there is often a halo problem due to the fact that the blue light and the re-emitted yellow light do not mix everywhere uniformly;
- the second consists in combining an LED emitting in the ultraviolet with at least one phosphorescent compound, each re-emitting light in a given colour. An example of a white LED using this technology is given in document **US 6 084 250**. For example, an LED emitting

between 370 and 410 nm is combined with a mixture of phosphorescent compounds, namely $Eu:BaMgAl_{11}O_{17}$, Cu:ZnS and YVO_4. This type of LED gives a white light having a good CRI, close to that of fluorescent lamps; and

- the third consists in mixing the colours emitted by several colour LEDs so as to obtain a white light. This technique has the advantage of being more flexible as it does not require the use of phosphorescent compounds. It is possible, by acting separately on the intensity of the LEDs, for the colour temperature to be finely adjusted, thereby making it possible to obtain a whole range of white lights (ranging from "hot" white to cold blue-white). In practice, to obtain white light, it is necessary for the LEDs to emit monochromatic light corresponding the sensitivity maxima of each type of cone present in the human eye (450 nm in the case of blue, 550 nm in the case of green and 620 nm in the case of red). If LEDs emitting at this precise frequency are not available, compensation is necessary by adjusting the emission intensity.

The emission spectra of white LEDs are broader than those of colour LEDs (an example of a spectrum is given in Figure 3). In the case of white LEDs, the scattering plastic according to the invention makes it possible to eradicate the drawbacks associated with the three technologies, for example the halo phenomenon associated with the first technology or else the imperfect colour mixing of the third technology. The scattering plastic according to the invention therefore applies particularly well for the production of luminous devices incorporating one or more white LEDs.

Scattering particles

To scatter the light emitted by the LED, the scattering particles must have a mean diameter of between 0.5 and 100 μm, advantageously between 1 and 80 μm and preferably between 1 and 70 μm. The particle size distribution is relatively narrow, depending on the type of particle and depending on the method of obtaining the particles. Preferably, the distribution covers the range from 0.01 to 200 μm, that is to say 90% of the particles have a size between 0.01 and 200 μm.

It is also necessary for the difference between the refractive indices (measured according to ASTM D 542) of the scattering particles and of the transparent plastic to be greater than 0.02 and preferably between 0.02 and 1.

The scattering particles may be of organic or mineral type and may advantageously be chosen from the families described below. The content of scattering particles is by weight between 3 and 30%, preferably between 5 and 30%, most preferably between 5 and 10% (wt% relative to the transparent plastic).

Polyamide particles

Among organic scattering particles, mention may be made of polyamide particles such as those described, for example, in Application **EP 0 893 481**. As polyamide, it is possible to use those obtained from lactams, such as for example ε-caprolactam, oenantholactam and undecano-lactam, or from amino acids having from 4 to 20 carbon atoms, such as aminocaproic, 7-aminoheptanoic, 11-aminoundecanoic and 12-aminododecanoic acids. It is also possible to use products resulting from the condensation of diamines, such as hexamethylenediamine, dodecamethylenediamine, metaxylylenediamine and trimethylhexamethylenediamine, with dicarboxylic acids, such as isophthalic, terephthalic, adipic, suberic, azelaic, sebacic, dodecanedioic and dodecane-dicarboxylic acids. For example, mention may be made of the polyamides nylon-6,6, nylon-6,9, nylon-6,10 and nylon-6,12, which are products resulting from the condensation of hexamethylenediamine with adipic acid, azelaic acid, sebacic acid and 1,12-dodecanedioic acid, respectively, or else nylon-9,6, which is a product resulting from the reaction of nonamethylenediamine with adipic acid.

Among these polyamides, mention may most particularly be made of nylon-6, obtained by the polymerization of ε-caprolactam, nylon-11, obtained by the condensation of 11-aminoundecanoic acid, and nylon-12, obtained by the polycondensation of 12-aminododecanoic acid or dodecanolactam.

The polyamide particles may be obtained by milling granules or by hot dissolution in suitable solvents followed by precipitation and cooling. It is also possible to obtain polyamide particles directly by anionic polymerization, as taught in documents **EP-B1-192 515** or **EP-B1-303 530.** The polyamide powder obtained by the method described in **EP-B1-303 530** is particularly preferred.

Suitable polyamide particles are for example those sold by Arkema under the names RILSAN or ORGASOL. For example, they may be particles of ORGASOL® 2001 EX D NAT 1 (nylon-12 particles with a mean diameter of 10 µm ± 1.5 µm), ORGASOL® 2001 UD NAT 1 (nylon-12 particles with a mean diameter of 5 µm ± 1 µm) or ORGASOL® 3501 EX D NAT 1 (6/12 copolyamide particles with a mean diameter of 10 µm ± 3 µm), and a melting point of 142 ± 2°C).

PTFE particles

Among organic scattering particles, mention may be made of polytetrafluoroethylene (PTFE) particles such as those described in Internal Application **WO 01/66644.** These particles may be obtained by a suspension or emulsion polymerization process. Suitable PTFE particles are for example those sold by DuPont under the name ZONYL. For example, it may be ZONYL 1000 (PTFE with a mean diameter of 11 μm) or ZONYL 1200 (PTFE with a mean diameter of 4 μm). The refractive index of PTFE is 1.376 according to the Polymer Handbook published by Wiley Interscience.

One advantage of PTFE particles is that they possess a high melting point (around 320°C) so that they can be introduced into the transparent plastic in the melt state, that is to say at high temperatures, possibly above 250°C.

Crosslinked styrene-based particles

Among organic scattering particles, mention may be made of particles of a crosslinked polymer comprising at least 50% styrene and 0.5 to 20%, preferably 1 to 10%, of a monomer possessing at least two double bonds C=C acting as crosslinking agent. This may for example be 1,4-butanediol di(meth)acrylate, ethylene glycol di(meth)acrylate, tetraethylene glycol di(meth)-acrylate, propylene glycol di(meth)acrylate, penta-erythritol tetra(meth)acrylate, allyl methacrylate or divinylbenzene.

The styrene-based crosslinked polymer advantageously comprises 0 to 20% of a comonomer having at least one ethylenic unsaturation copolymerizable with styrene, chosen for example from chlorostyrene, bromostyrene, vinyltoluene, acrylonitrile, alpha-methylstyrene or a C_1-C_{10} alkyl(meth)acrylate such as, for example, methyl (meth)acrylate, ethyl (meth)acrylate, butyl (meth)-acrylate, benzyl (meth)acrylate, 2-hydroxyethyl (meth)-acrylate, phenyl (meth)acrylate, etc. Chlorostyrene and bromostyrene are the two monomers of choice for modifying the refractive index of the styrene-based crosslinked particles.

Crosslinking makes it possible to prevent the particles from deforming when they are incorporated into the transparent plastic.

The styrene-based scattering particles are advantageously prepared by polymerization in a dispersed medium, such as suspension, emulsion or microsuspension polymerization. The styrene-based scattering particles are substantially spherical. The mean diameter is determined by parameters known to those skilled in the art, such as, for example the stirring rate or the amount of suspension agent.

Suitable styrene-based scattering particles are for example those sold by Sekisui under the name SBX. They may be for example SBX-6, SBX-8, SBX-12 or SBX-17 particles (styrene-based crosslinked particles with a mean diameter of 6, 8, 12 or 17 μm). The refractive index of the SBX particles is 1.59 (according to the information found on the Internet site of the Sekisui group).

Methyl-methacrylated-based crosslinked particles

Among organic scattering particles, mention may be made of particles of a crosslinked polymer comprising at least 50% methyl methacrylate and 0.5 to 20%, preferably 1 to 10%, of a monomer possessing at least two C=C double bonds acting as crosslinking agent. This may for example be 1,4-butanediol di(meth)acrylate, ethylene glycol di(meth)acrylate, tetraethylene glycol di(meth)acrylate, propylene glycol di(meth)acrylate, pentaerythritol tetra(meth)acrylate, allyl methacrylate or divinylbenzene.

The crosslinked polymer based on methyl methacrylate advantageously includes from 0 to 20% of a comonomer having at least one ethylenic unsaturation copolymerizable with methyl methacrylate, chosen from styrene, alpha-methylstyrene, acrylonitrile, a C_1-C_{10} alkyl(meth)acrylate, such as for example methyl acrylate, ethyl (meth)acrylate, butyl (meth)acrylate, benzyl (meth)acrylate, 2-hydroxyethyl (meth)acrylate and phenyl (meth)acrylate. Styrene, α-methylstyrene, benzyl methacrylate and phenyl methacrylate are monomers of choice for modifying the refractive index of the methyl-methacrylate-based particles.

The methyl-methacrylate-based scattering particles are advantageously prepared by polymerization in a dispersed medium, such as suspension polymerization, according to a recipe given for example in the document **EP 1 022 115, US 2002/0123565** or **US 2002/0123563.** The methyl-methacrylate-based scattering particles are substantially spherical. The mean diameter is determined by parameters known to those skilled in the art, such as for example the stirring speed or the amount of suspension agent.

The following examples describe methyl-methacrylate-based scattering particles that are obtained by suspension polymerization:

Example 1

- 95% methyl methacrylate; 4% ethyl acrylate; 1% allyl methacrylate;
- mean diameter: 30 µm, with 90% of the particles having a mean diameter of less than 40 µm;
- refractive index n_D: 1.4935 (according to ASTM D 542).

Example 2

- 74% methyl methacrylate; 25% styrene; 1% allyl methacrylate;
- mean diameter: 30 µm, with 90% of the particles having a mean diameter of less than 40 µm;
- refractive index n_D: 1.5217 (according to ASTM D 542).

Example 3

- 55% methyl methacrylate; 40% benzyl methacrylate; 5% allyl methacrylate;

- mean diameter: 7 µm, with 90% of the particles having a mean diameter of less than 10 µm.

Silicone particles

Among organic scattering particles, mention may also be made of silicone-based particles. These particles are obtained by milling a silicone until scattering particles are obtained that have the size sufficient to be dispersed in the transparent plastic and to correctly scatter the light emitted by the LED. The silicone is obtained by the hydrolysis and condensation of chlorosilanes, such as dimethyldichlorosilane, diphenyldichlorosilane, phenylmethyldichlorosilane, trimethyltrichlorosilane or phenyltrichlorosilane. Preferably, the silicone will have been crosslinked by means of a free radical generator, such as for example benzoyl peroxide, para-chlorobenzoyl peroxide, *tert*-butyl peroxide or *tert*-amyl peroxide.

Suitable scattering silicone particles are for example those sold by Toray Dow Corning Silicone under the name TORAY-FIL DY33-719. These particles have a refractive index of 1.42 and a mean size of 2 µm.

Mineral particles

Among mineral scattering particles, mention may be made of $BaSO_4$, TiO_2, ZnO, $CaCO_3$, MgO and Al_2O_3 particles. Mineral scattering particles having a mean diameter of between 0.5 and 15 µm, preferably between 0.5 and 10 µm, are preferred. The advantage of mineral scattering particles over organic scattering particles is that it is possible to obtain good light scattering with a smaller amount. Typically, the content of mineral scattering particles is between 3 and 15%, advantageously between 3 and 10% and preferably between 5 and 10%. The mineral scattering particles are preferably colourless.

Another advantage of mineral scattering particles is associated with their thermal stability. This is because, if the scattering particles are mixed with the transparent plastic in the melt state, it is necessary to reach high values in order to melt the plastic and facilitate homogenization. For example, in the case of PMMA, homogenization is carried out at temperatures between 210 and 300°C. In the case of polycarbonate, the extrusion temperatures may exceed 300°C. The organic scattering particles are liable to degrade at high temperature and result in yellowing/blackening of the particles. This detracts from the final appearance of the compound.

From the mineral scattering particles it is advantageous to choose $BaSO_4$ particles (1.64 refractive index n_D) as they scatter the light very well, are very easily dispersed in the transparent plastic and are perfectly colourless. In the case of the process for manufacturing cast PMMA, $BaSO_4$ particles are dispersed very well in the composition to be cured and do not aggregate therein. As an example, Blanc Fixe N particles sold by the German company Sachtleben Chemie may be used.

Other mineral particles are hollow glass microspheres, as described in International Application **WO 03/072645.** For example, glass microspheres sold under the name SCOTCHLITE® by 3M (Minnesota Mining and Manufacturing Company) may be suitable. These microspheres are obtained by known methods. According to **EP 1 172 341,** it is possible to add, for example, an inflammable liquid to a compound containing ingredients suitable for the preparation of glass and a blowing agent, and then to subject the entire composition to wet milling so as to obtain a dispersion of particles having a mean size of 3 µm. This slurry is then atomized by means of a two-fluid nozzle, at a pressure of between 0.2 and 2 MPa, so as to form droplets. Heating these droplets results in the formation of hollow glass microspheres.

As ingredients suitable for preparing the glass, mention may be made of silica sand, volcanic ash, perlite, obsidian, silica gel, zeolite, bentonite, borax, boric acid, $Ca_3(PO)_4$, Na_2SO_4, $Mg_4P_2O_7$, Al_2O_3, a compound deriving from SiO_2, B_2O_3 or Na_2O. It is preferred to use a glass based on borosilicates, especially a soda-lime glass. The blowing agent generates gas when the mix of ingredients resulting in the glass undergoes vitrification by heating, and has the effect of producing substantially spherical hollow particles of molten vitrified glass. The blowing agent may for example be sodium, potassium, lithium, calcium, magnesium, barium, aluminium or zinc sulphate, carbonate, nitrate or acetate.

Dyes

It is possible to add one or more dyes or pigments to the transparent plastic in order to obtain a coloured or tinted cover. A person skilled in the art knows how to choose the dye(s) or pigment(s) needed to obtain a given colour or tint, being characterized by given x/y coordinates. As examples of dyes and pigments, mention may be made of the following: copper phthalocyanine green, copper phthalocyanine blue, ultramarine blue, pyrazolone yellow, diketopyrrolopyrrole red, red iron oxide, ultramarine, chromium-titanium yellow, and dyes of the anthraquinone type.

Examples

- PMMA cover having a yellow colour obtained using 0.08 to 0.09% of pyrazolone yellow;
- PMMA cover having a green colour obtained using 0.01 to 0.02% of copper phthalocyanine; and
- PMMA cover having a red colour, obtained using 0.06 to 0.07% naphthol AS.

Surface roughness

At least one of the faces of the cover may have a relatively pronounced surface roughness (producing what is often called a "frosted" effect). The surface roughness may be obtained in several ways. According to a 1st method, in the case of a cast sheet, the glass mould, which forms the sheet, has itself a surface roughness that has been obtained by treating the glass of the mould, for example with hydrofluoric acid. According to a 2nd method, sandblasting may be used as taught in document **WO 03/083564** or **US 3 497 981**. The surface roughness, denoted by Ra, is expressed in microns and can be measured using a roughness meter (for example of the Talysurf Surtronic 3P brand from Rank-Taylor-Hobson) according to the ISO 4287 and ISO 4288 standards. The value of the surface roughness Ra is of the order of a few μm. This value is between 0.5 and 4 μm, preferably between 1 and 3 μm.

The surface roughness on at least one of the faces of the cover allows the light-scattering effect of the scattering particles to be enhanced.

The faces of the cover according to the invention may also be perfectly smooth and not have any pronounced roughness. In this case, the value of the surface roughness Ra is less than 400 nm, advantageously less than 300 nm and preferably less than 100 nm.

Method of obtaining the cover

There are several methods known to those skilled in the art for producing the cover of the luminous device of the invention. The scattering particles and optional other additives (dye(s), impact modifier, UV stabilizer, antioxidant, etc.) are mixed with the transparent plastic by means of an extruder or any other mixing tool suitable for thermoplastics as known to those skilled in the art. Recovered at the exit of the extruder are granules that are then formed to the desired shape using a conversion technique for thermoplastics, for example injection moulding or compression moulding. It is also possible to adapt the extruder in order to produce a sheet. The cover is then obtained after forming the sheet to the desired shape after cutting and/or thermoforming.

In the case of PMMA, it is also possible to use the cast process to manufacture the sheet (then referred to as a cast PMMA sheet). This process consists in using a mould formed from two flat plates, for example made of inorganic glass, which are separated by a peripheral seal, generally made of polyvinyl chloride, for sealing between the two plates. The mould is closed by clamps placed along the sides. The composition to be polymerized (i.e. the methyl methacrylate and optionally the comonomer(s)), to which a polymerization initiator in sufficient amount has been added, is poured into the mould. The composition to be polymerized contains the scattering particles and all the other optional additives (for example demoulding agent, chain transfer agent for controlling the average molecular weight of the final polymer, antioxidant, UV stabilizer, dye(s), etc.). The polymerization is carried out or completed, depending on the case, by placing the mould in water (a process called "liquid pool" polymerization) or in an oven at the necessary temperature (40-80°C), and then in an oven (at about 100-130°C) for the post polymerization. The thickness of the seal determines the thickness of the polymer sheet obtained.

The cover may have all kinds of geometries, depending on the nature of the intended application. For example, it may be in the form of a flat, curved or domed sheet, whether rectangular or circular, in the form of a disc, etc. It may also take the form of a letter of the alphabet or of any other sign or symbol in the case of an illuminated sign, as illustrated for example in Figure 5. Examples of other shapes are also given in the following documents: **US 2004/0255497, FR 2 857 434, EP 1 402 504 and US 2005/0039361.**

The cover has a thickness of between 0.1 and 15 cm, but is still between 0.1 and 10 cm, advantageously between 0.1 and 7 cm, preferably between 0.1 and 5 cm and even more preferably between 0.2 and 4 cm.

Luminous device

The luminous device according to the invention comprises at least one LED, the light flux of which is greater than 3 Lm, advantageously greater than 5 Lm, preferably greater than 10 Lm, more preferably greater than 50 Lm, and at least one cover made of a transparent plastic in which 3 to 30% of scattering particles are dispersed. The cover makes it possible to:

- ensure transmission of the light emitted by the LED(s);
- mask and protect the LED(s);
- provide a uniform and nondazzling illumination;
- reduce the directional lighting effect of the LED(s); and
- reduce, or even eliminate the drawbacks of LEDs, especially the halo effects in white LEDs.

The cover is preferably in the form of a single layer and is not obtained by coextrusion of two or more layers as in **EP 1369224 A1**.

Advantageously, the luminous flux is between 3 and 200 Lm, better still between 3 and 100 Lm, preferably between 5 and 100 Lm, even more preferably between 10 and 100 Lm and very preferentially between 50 and 100 Lm. This luminous flux can be measured using the method recommended by the CIE (International Commission on Illumination) in Publication 127 (1997).

Thanks to LEDs having a high luminous flux, it is possible to reduce the number of LEDs in order to obtain a given illumination. Compared with an incandescent lamp or a neon tube, it is possible to obtain luminous devices that are more compact and consume less electrical power.

It may be a colour LED or a white LED.

A colour LED may be combined with a colourless cover or better still a coloured cover. To obtain the best illumination effect, it is preferable to combine a coloured cover with a colour LED and it is preferable for the colour of the cover to be as close as possible to that emitted by the LED. A more mathematic interpretation may be given by using the x/y chromaticity diagram, which allows any colour to be represented. Thus, relative to the straight line D passing through the achromatic point A of coordinates $x_A = 1/3$ and $y_A = 1/3$ and through the point B of the colour LED of coordinates x_B and y_B, the light transmitted by the coloured cover, represented in the chromaticity diagram by the point T, must preferably lie on the line D at 0.2 x/y units, preferably 0.1 x/y units, from the point B and along a line perpendicular to D, at 0.05 x/y units, preferably 0.03 x/y units, from the point B. The point T must therefore lie inside a rectangle having the point B as orthocentre. This rectangle has a half-length L that corresponds to 0.2 x/y units, preferably 0.1 x/y units, and a half-width l that corresponds to 0.05 x/y units, preferably 0.03 x/y units.

Red LED

A red LED may emit between 610 and 640 nm. For example, it is possible to envisage using a SUPERFLUX HPWT-RD00 or HPWT-MD00 red LED sold by Lumileds, which has a luminous flux of 3.5 Lm.

It may also be the D 001 RGB 24 V red LED emitting between 619 and 629 nm, sold by Tridonic & co, which has a luminous flux of 23.6 Lm. Another example is the red LED P 511 R emitting between 624 and 630 nm, sold by Tridonic & co, which has a minimum luminous flux of 135 Lm.

Green LED

A green LED may emit between 500 and 550 nm. For example, it is possible to envisage using an HPWN-MG00-0000 green LED sold by Lumileds, which has a luminous flux of 3.0 Lm.

Yellow LED

A yellow LED may emit between 570 and 610 nm.

Blue LED

A blue LED may emit between 440 and 500 nm.

It is also possible to combine a white LED with a colourless cover or with a coloured cover. The following examples of coloured covers may be given. In these examples, the three values L, a*, b* are used to characterize the principal colour in the CIELAB system. L denotes the luminosity and extends from 0 (black) to 100 (white). The value a* measures the red and green of the colour: the colours tending toward green have a negative a* value while those tending toward the red have a positive a* value. The b* value measures the blue and the yellow of the colour: colours tending toward the yellow have a positive b* value while those tending toward the blue have a negative b* value. The L, a*, b* values are measured using a colorimeter (especially according to the ASTM E 308 standard).

Examples

- a PMMA cover having a luminosity L of 23.7, an a* value of 0.3 and a b* value of -0.8;
- a PMMA cover having a luminosity L of 35.5, an a* value of -0.5 and a b* value of -1.5;
- a PMMA cover having a luminosity L of 34.1, an a* value of -18.8 and a b* value of -0.1;

- a PMMA cover having a luminosity L of 34.6, an a* value of 3.1 and a b* value of -32.7; and
- a PMMA cover having a luminosity L of 33.2, an a* value of 8.5 and a b* value of -28.2.

Preferably, a white LED is combined with a colourless cover.

Examples of white LEDs

It is possible, for example, to envisage using a white LED of the LUXEON STAR type sold by Lumileds. It is also possible to envisage using a white LED sold by Tridonic & co under the reference Daylight P115-BL, which has a luminous flux of at least 27.7 Lm.

The cover is separated from the LED(s) by a distance of between 1 and 50 cm, better still between 2 and 50 cm, preferably between 2 and 20 cm and even more preferably between 3 and 20 cm. The luminous device is distinguished from edge-emitting devices, as described for example in **EP 0 893 481**. This is because, in the luminous device according to the invention, it is not the edge of the cover that is illuminated but one of the faces of the cover.

[Uses]

The luminous device according to the invention has a variety of applications such as, for example:

- interior lighting (living room lamps, office lamps, etc.);
- advertising displays;
- direction lighting or escape route marking;
- illuminated signs (in this case, the cover may especially have the form of a letter, a number, a symbol or any other sign);
- traffic signalling; and
- automobile lighting (for example the luminous device may be a headlamp, a daytime light, a direction indicator, a stop light, a fog lamp,

reversing light, etc.)

The invention also relates to these uses.

[examples]

Several cast sheets in PMMA containing different amounts of particles of $BaSO_4$ (scattering particles from Sachtleben Chemie, average size 8 µm) and having a thickness of 3 mm were prepared according to common techniques. The sheets were used as covers for LEDs of luminous flux, ones of flux lower than 5 Lm and others of flux higher than 5 Lm. The distance between the cover sheet and the top of the LED was also varied. The LED is positioned in front of the surface of the sheet and not along its edge.

LEDs of flux < 5 Lm

OS LM 03 A-A red

OS LM 03 A-T green

OS LM 03 A-B blue

OS LM 03 A-W white

OS LM 03 A-Y yellow

All these LEDs are from OSRAM.

LEDs of flux > 5 Lm

Luxeon star LXHL-LM1D white

Luxeon star LXHL-LM3C green : minimum flux 50 Lm (700 mA, junction temperature : 25°C)

Luxeon star LXHL-LB3C blue : minimum flux 20 Lm (700 mA, junction temperature : 25°C)

Luxeon star LXHL-LH3C red : minimum flux 120 Lm (1400 mA, junction temperature : 25°C)

Luxeon star LXHL-ML1D amber

All these LEDs are from LUMILED.

Table I gives the visual observations of the luminous devices that associate the LEDs and the sheets as the distance between the LED and the surface of the LED is varied.

Table I

sheet	amount of $BaSO_4$ (wt%)	LED with a luminous flux < 5 Lm			LED with a luminous flux > 5 Lm		
distance sheet / top of the LED		3 cm	6 cm	9 cm	3 cm	6 cm	9 cm
A	3%	≈	+	+	-	-	-
B	3.5%	≈	+	+	-	-	-
C	5.5%	+	+	≈	-	≈	≈/+
D	5.7%	+	+	≈	-	≈	≈/+
E	7%	+	+	≈	+	+	+
F	7.5%	+	+	≈	+	+	+

<u>observations</u>

\+ good visual effect

\- unsufficient visual effect (poor diffusing properties and/or uneveness of the illumination)

≈ debatable effect (between + and -)

The luminous device associating sheet F and a low power LED is not bright enough when the distance exceeds 6 cm whereas for the high power LEDs, the distance is less critical. For sheets E and F, the luminous devices provide an even and correct illumination (without dark areas or hot spots).

Claims

1. Luminous device comprising at least one LED having a luminous flux of greater than 3 Lm, advantageously greater than 5 Lm, preferably greater than 10 Lm and even more preferably greater than 50 Lm, and a cover made of a transparent plastic in which scattering particles with a content by weight of 3 to 30%, advantageously 5 to 30%, most preferably of 5 to 10% are dispersed.

2. Luminous device according to Claim 1, characterized in that the transparent plastic is of the thermoplastic or thermosetting type and has a light transmission in the visible range of at least 50%, preferably at least 70% and even more preferably at least 80% according to the DIN 67-507 standard.

3. Luminous device according to either of Claims 1 and 2, characterized in that the transparent plastic is chosen from crystal polystyrene, polyethylene terephthalate, a transparent, especially clarified, polyolefin, clarified polypropylene, PMMA, a transparent polyamide or polycarbonate.

4. Luminous device according to either of Claims 1 and 2, characterized in that the transparent plastic is a methyl methacrylate homopolymer or a copolymer comprising from 80 to 99.5% by weight of methyl methacrylate and from 0.5 to 20% by weight of at least one monomer having at least one ethylenic unsaturation that can copolymerize with methyl methacrylate.

5. Luminous device according to Claim 4, characterized in that the transparent plastic is a copolymer comprising, by weight, 90 to 99.7%, preferably 90 to 99.5%, of methyl methacrylate and 0.3 to 10%, preferably 0.5 to 10%, of at least one monomer having at least one ethylenic unsaturation that can be

copolymerized with methyl methacrylate.

6. Luminous device according to either of Claims 1 and 2, characterized in that the transparent plastic is a polyester obtained by the reaction of at least one carboxylic acid derivative with at least one aromatic or aliphatic diol.

7. Luminous device according to Claim 6, characterized in that the polyester is a homopolycarbonate or a copolycarbonate based on a bisphenol of formula HO-Z-OH for which Z denotes a divalent organic radical possessing from 6 to 30 carbon atoms and containing one or more aromatic group(s).

8. Luminous device according to Claim 7, characterized in that the diphenol is chosen from dihydroxydiphenyls, bis(hydroxyphenyl)alkanes, bis(hydroxyphenyl)cycloalkanes, indane bisphenols, bis(hydroxyphenyl) ethers, bis(hydroxyphenyl) ketones, bis(hydroxyphenyl) sulphones, bis(hydroxyphenyl) sulphoxides and α,α'-bis(hydroxyphenyl)diisopropylbenzenes.

9. Luminous device according to one of the preceding claims, characterized in that the scattering particles have a mean diameter of between 0.5 and 100 μm, advantageously between 1 and 80 μm and preferably between 1 and 70 μm.

10. Luminous device according to one of the preceding claims, characterized in that the difference between the refractive indices (ASTM D 542) of the scattering particles and the transparent plastic is greater than 0.02 and preferably between 0.02 and 1.

11. Luminous device according to one of Claims 1 to 10, characterized in that the scattering particles are polyamide particles, PTFE particles, crosslinked

styrene-based particles, methyl-methacrylate-based crosslinked particles or silicone particles.

12. Luminous device according to one of Claims 1 to 10, characterized in that the scattering particles are $BaSO_4$, TiO_2, ZnO, $CaCO_3$, MgO or Al_2O_3 particles or hollow glass microspheres.

13. Luminous device according to Claim 12, characterized in that the mean diameter of the mineral scattering particles is between 0.5 and 15 μm, preferably between 0.5 and 10 μm.

14. Luminous device according to either of Claims 12 and 13, characterized in that the content of scattering particles is between 3 and 15%, advantageously between 3 and 10% and preferably between 5 and 10%.

15. Luminous device according to one of Claims 1 to 14, characterized in that at least one of the faces of the cover has a roughness Ra of between 0.5 and 4 μm, preferably between 1 and 3 μm.

16. Use of the luminous device according to any one of Claims 1 to 15 for :

- interior lighting;
- advertising displays;
- direction lighting or escape route marking;
- illuminated signs ;
- traffic signalling; and
- automobile lighting.

17. Use according to Claim 16, characterized in that the cover has the form of a letter or a number.

18. Use according to Claim 16, characterized in that the automobile lighting is a headlamp, a daytime light, a direction indicator, a stop light, a fog lamp or a reversing light.

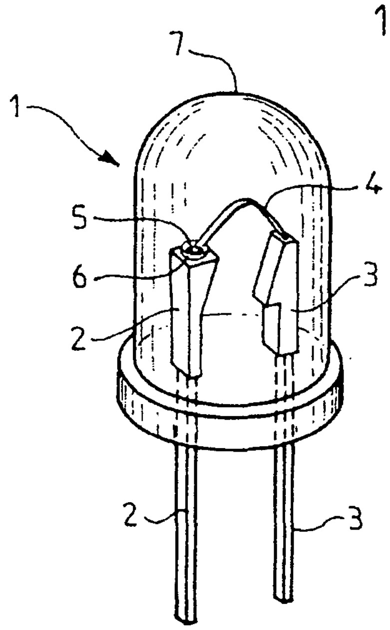


FIG.1

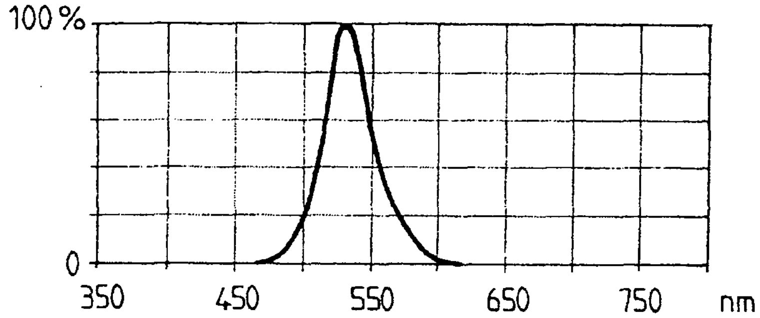


FIG. 2

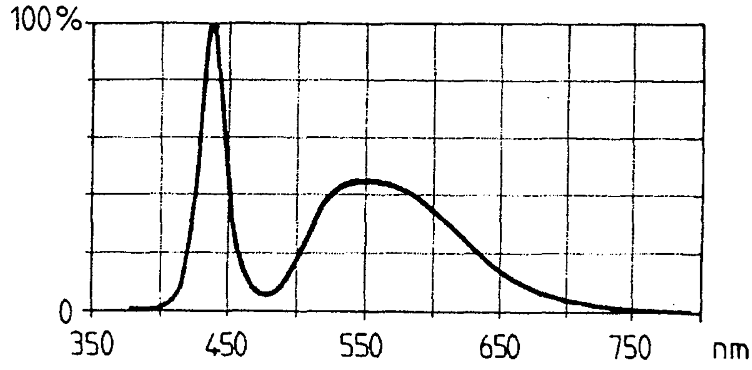


FIG. 3

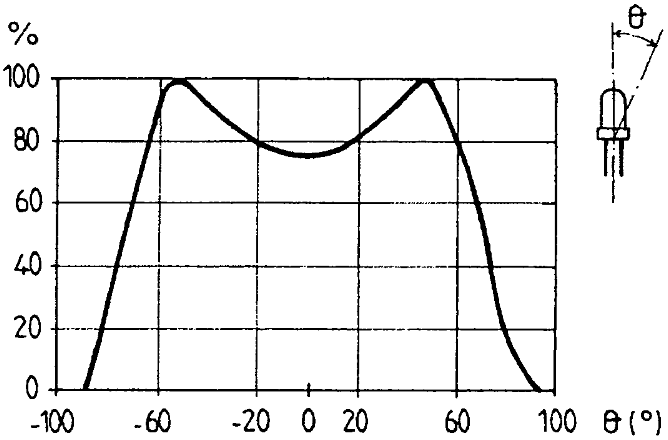


FIG. 4

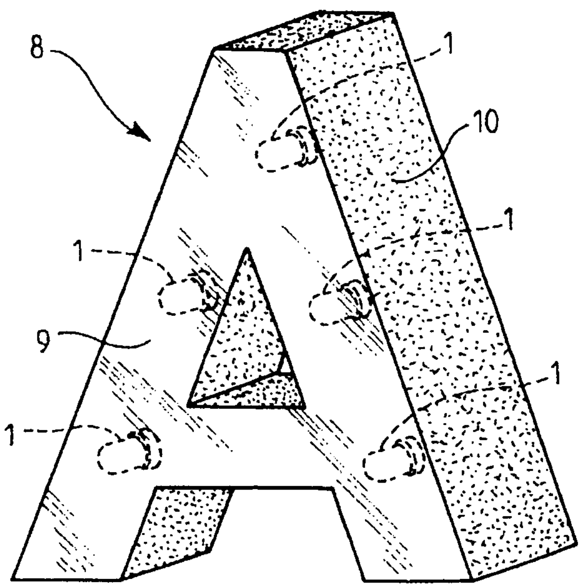
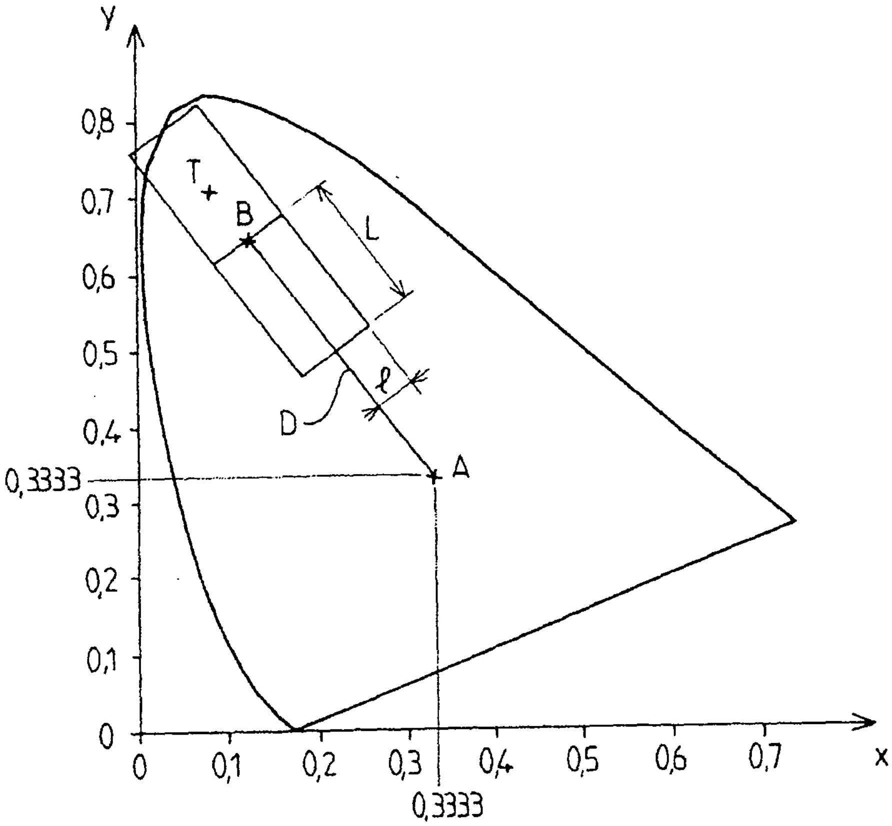


FIG.5


y
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0,6
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FIG.6