

PATENT SPECIFICATION

NO DRAWINGS

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(54) IMPROVEMENTS IN AND RELATING TO THE VAPOUR DEPOSITION OF LAYERS ON CURVED OPTICAL SURFACES

(71) We, BALZERS PATENT- UND BETEILIGUNGS - AKTIENGESSELLSCHAFT, a Liechtenstein joint-stock company, of FL-9496 Balzers, Principality of Liechtenstein, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The present invention relates to the deposit-of layers from the vapour phase on curved optical surfaces.

15 In the vacuum vapour deposition on curved optical surfaces, e.g. in the production of concave mirrors, it is a difficult problem to deposit on all parts of these surfaces layers of the prescribed thickness. In the Patent Specification No. 1,088,629 a solution of this problem has been disclosed which consists in that when depositing from the vapour phase the distance between the substrate to be deposited upon and the vapour source at a residual gas pressure between 0.5×10^{-3} Torr. and 1.5×10^{-3} Torr. is so dimensioned, that this distance is larger than the mean free path length of the gas molecules, the residual gas being formed by argon, crypton or xenon or mixtures of these gases, and the substrate to be deposited upon is subjected to a heat treatment after the deposition of all the layers provided. The effectiveness of this known solution of the problem is based on the scatter of the molecules of the substance to be deposited from the vapour phase by the molecules or atoms or the residual gas in the vapour deposition chamber, whereby the adverse effect of the different distances of the individual parts of the curved surface to be deposited upon from the vapour source, and of the different angles of incidence of the vapour beams on the individual parts of the surface are compensated to a far reaching extent.

45 The disadvantage of this known method consists in that rare gases are expensive. However, when attempting to use other gases for scattering the vapour molecules, it has

been found, that frequently the possible reactions between the latter and the residual gas have an adverse effect. For example by oxygen oxides, by nitrogen nitrides and by other gases other compounds are formed and embedded in the layers (at pressures above 10^{-4} Torr even sometimes only these compounds are obtained). By the embedding of molecules of the residual gas and the compounds thereof in the layer the structure of the layer is disturbed and thus the adhesion of the layers on the base and their own strength (hardness) are reduced. By a subsequent heat treatment the atoms of rare gases possibly embedded in the layer can be expelled and the layer thus be "healed". However, when using another gas for the scattering the compounds formed cannot be removed from the layer, and possible chemical reactions between the embedded molecules of the residual gas and the material of the layer may even be promoted, i.e. initiated or accelerated, by the heat treatment.

It has been found, that layers deposited under the action of a residual gas atmosphere from the vapour phase have often a non-permissible high scattering effect on light unsuitable for optical purposes, which is caused likewise by the embedded residual gas molecules and compounds thereof disturbing the structure of the layer. Losses in luminosity by the scattering of the light appear to be the larger, the higher is the molecular weight of the residual gas used for the vapour phase deposition, i.e. the opinion that heavy molecules are more suitable for the present purpose than light ones is not necessarily valid, as tests by the inventor have shown.

The present invention has the object of providing a method for the deposition on optical surfaces of layer-forming substances from the vapour phase *in vacuo* under the influence of residual gas, which is chemically inert thereto, which method is at least as effective as the known methods as regards

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the optical and mechanical properties of the layers produced and yet dispenses with the necessity of using expensive rare gases, and is consequently much more economic in production. It has been found, surprisingly, that there exists a non-rare gas, which allows to keep the advantages of vapour phase deposition in a rare gas atmosphere but to avoid the disadvantages usually occurring when depositing from the vapour phase in an atmosphere of non-rare residual gases. The method according to the present invention is characterised in that the vapour deposition is carried out in an atmosphere containing carbon dioxide. In doing so, as in the known method, the distance between the vapour source and the surface to be deposited upon is to be chosen larger than the mean free path length of the vapour molecules in the residual gas atmosphere.

Hereinafter the method according to the present invention will be described in more detail with reference to an embodiment by way of example:—For the production of a so-called cold light mirror on a concave mirror surface of a mirror body consisting of glass firstly a layer of magnesium fluoride of an optical thickness of $\lambda_1/2$ was deposited in a residual gas atmosphere of CO_2 at 3×10^{-4} Torr, and subsequently in the same CO_2 atmosphere nine layers of the thickness $\lambda_1/4$, alternately consisting of zinc sulphide and magnesium fluoride were deposited, followed by nine layers alternately consisting of ZnS and MgF_2 of the thickness $\lambda_2/4$ in the said residual gas atmosphere, wherein λ_1 and λ_2 denote two predetermined wave lengths, which for a usual cold light mirror may be chosen $\lambda_1 = 480 \text{ m}\mu$ and $\lambda_2 = 600 \text{ m}\mu$. All the thickness dimensions relate to the so-called optical layer thickness $s = nd$, wherein n is the refractive index of the layer, and d is the so-called geometrical layer thickness.

Obviously other layers systems having different layer thicknesses and layer numbers may be produced by the vapour deposition method described. In the aforesaid example layers of uniform thickness over the whole mirror surface were obtained, the distance of the mirror from the vapour source amounting to 40 cm, its radius of curvature to about 50 cm and the diameter of the mirror also amounting to 50 cm.

By varying the pressure of the residual gas atmosphere the distribution of the layer thicknesses may be influenced, namely by increasing the pressure an increase of the

layer thickness from the middle to the margin, and by reducing the pressure an inverse variation of thickness. In general, however, thicknesses uniform over the whole area are desired; the optimum pressure of the residual gas atmosphere for this purpose can readily be ascertained by a preliminary test.

In most cases it is advantageous to heat the bases to be deposited upon during the vapour deposition or subsequently.

The method according to the present invention has proved particularly advantageous in its application to magnesium fluoride and zinc sulphide as the substances to be deposited from the vapour phase. This may be caused by the particular chemical properties of these substances, and by their molecular weights. The said substances are known to be important starting materials for the production of thin layers for optical purposes. The method according to the present invention is however also applicable to other frequently used substances to be deposited from the vapour phase such as cryolite thorium fluoride and zinc selenide.

Possible arrangements of the vapour source and of carriers for the bases to be deposited upon from the vapour phase in a vacuum vapour deposition plant for carrying out the method according to the present invention are known to a person skilled in the art; one such arrangement is described in the Specification cited hereinabove.

WHAT WE CLAIM IS:—

1. A method for the deposition from the vapour phase *in vacuo* of layer forming substances on curved optical surfaces in a residual gas atmosphere containing carbon dioxide, wherein the distance between the vapour source and the surface to be deposited upon exceeds the mean free length of path of the vapour molecules in the residual gas atmosphere.
2. A method according to claim 1, wherein the vapour phase deposition is carried out at a CO_2 -pressure of between 10^{-4} and 10^{-3} Torr.
3. A method according to claim 1, wherein the layer-forming substances are magnesium fluoride and/or zinc sulphide.
4. A method according to claim 1, substantially as herein described.
5. A base having a curved optical surface when coated with layers by a method according to any of the preceding claims.

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2. A method according to claim 1, wherein the vapour phase deposition is carried out at a CO_2 -pressure of between 10^{-4} and 10^{-5} Torr.

3. A method according to claim 1, wherein the layer-forming substances are magnesium fluoride and/or zinc sulphide.

4. A method according to claim 1, substantially as herein described.

5. A base having a curved optical surface when coated with layers by a method according to any of the preceding claims.

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